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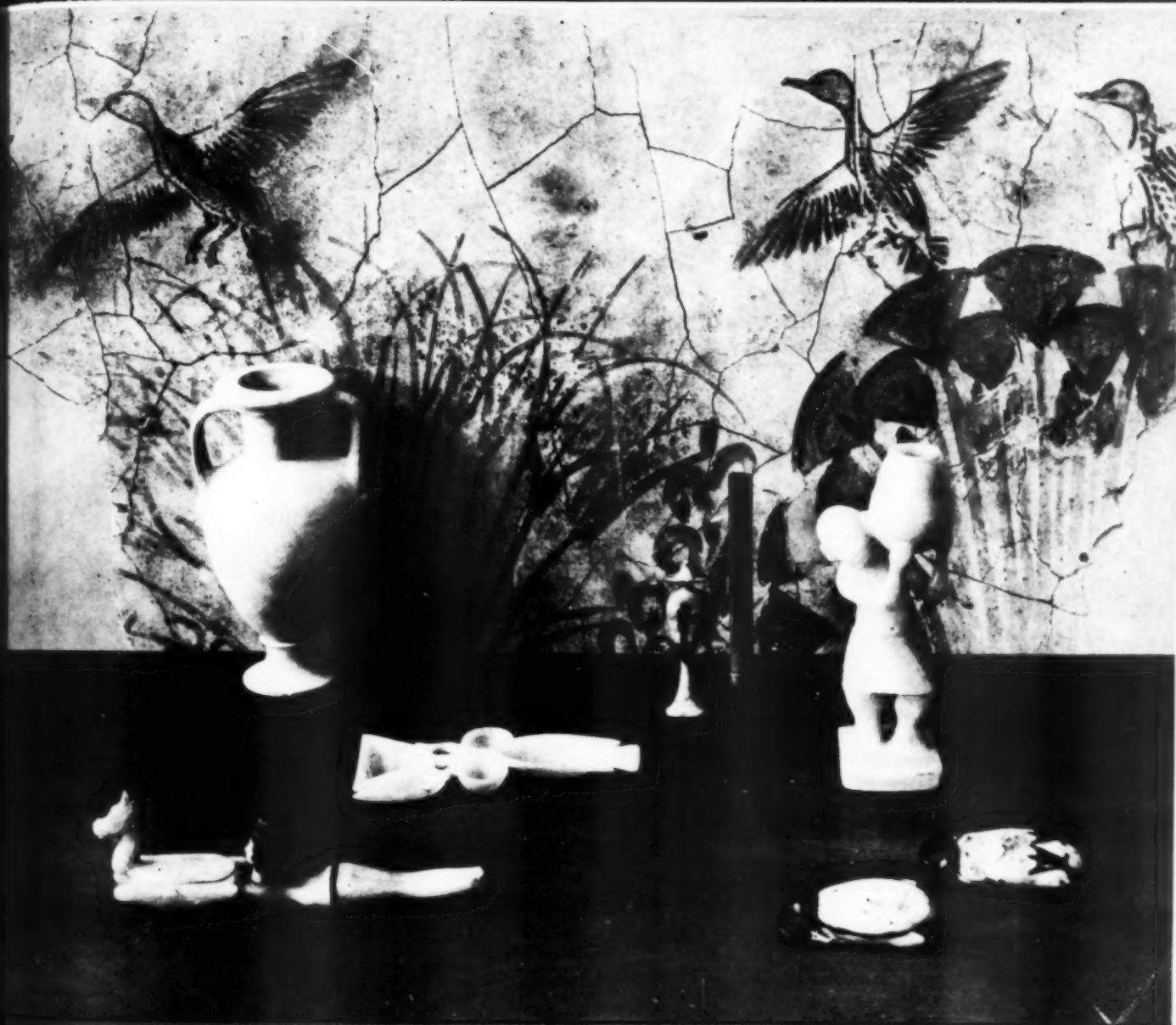
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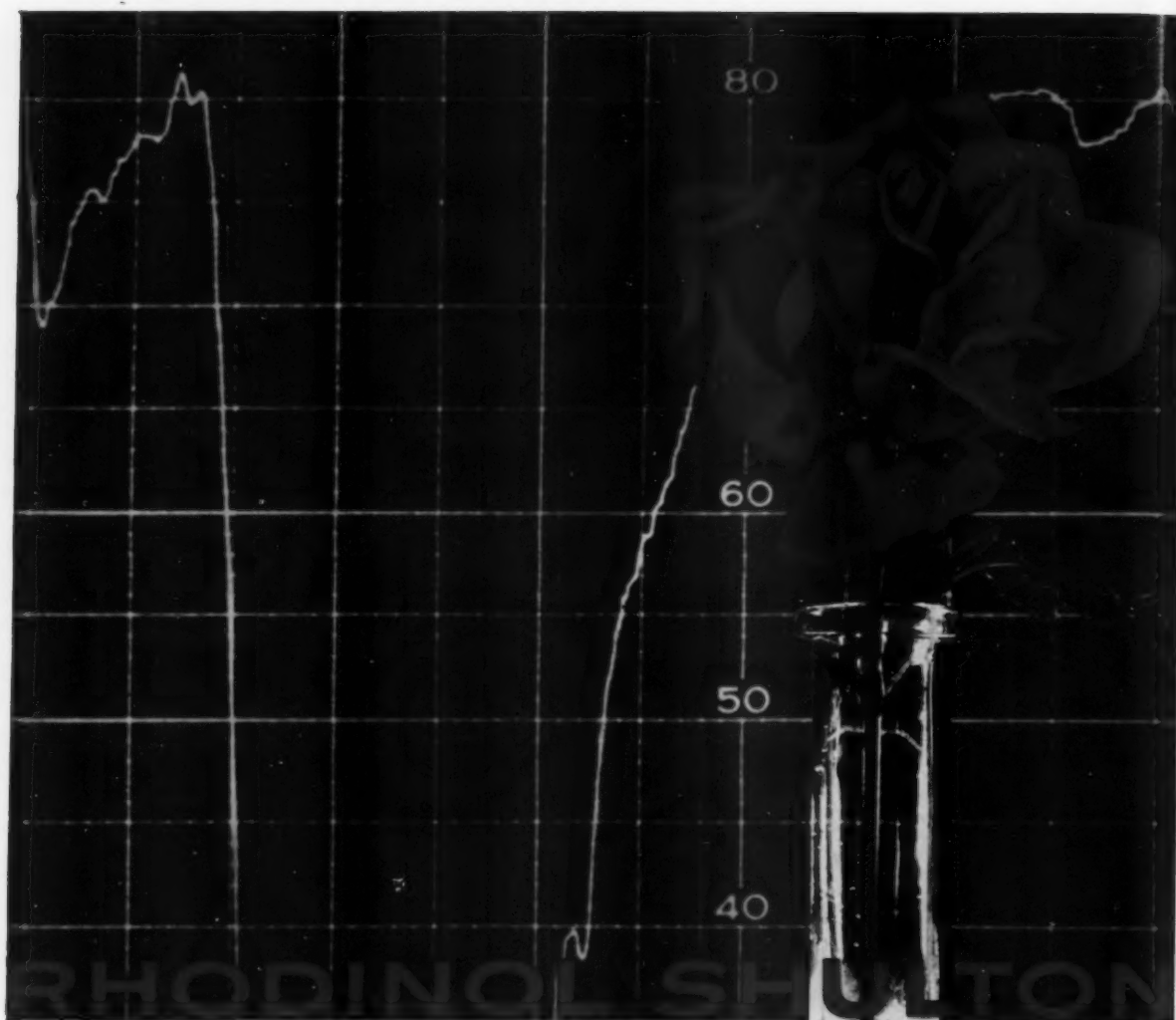
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MARCH 1960

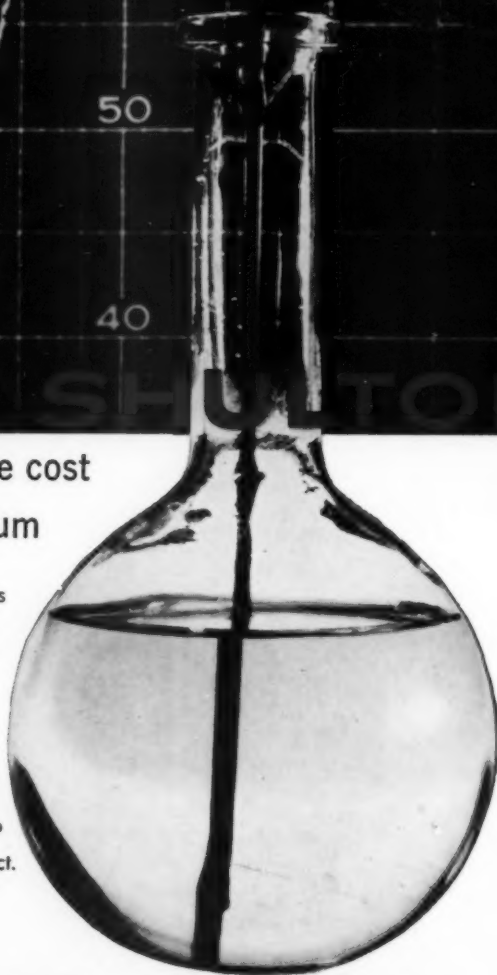
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American Perfumer AND AROMATICS

VOL. 75, NO. 3

MARCH, 1960

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New publisher of *American Perfumer*

With this issue the *American Perfumer* becomes the property of the Allured family, who have been publishers of business magazines for forty years. Our former publishing interest has been in the confectionery field, with *The Manufacturing Confectioner*, a monthly magazine, and two directories.

The addition of *American Perfumer* to our organization makes a very much stronger publishing organization, with two quite similar properties, and enough common interest to make them entirely compatible. The area of common interest is, of course, the very close relationship between confectionery flavors and perfume aromatics.

We are very optimistic about the future of *American Perfumer*, and the industry that it serves. For one thing, ours is a comparatively small organization in which *American Perfumer* will be a very important part. Therefore, it will receive the attention of our full staff, to provide the type of news and technical information that you have every reason to expect from the leading magazine in this field.

For another thing, the excellent staff that we inherited with the magazine will remain to guide its progress. M. G. deNavarre, our Editorial Director, has been functioning in this capacity brilliantly, witness the pace-setting Documentary series. This series will hit a high point with a *Perfume Documentary* in our June issue.

For many years, John Muller has been the primary industry contact for *American Perfumer* on both sales and news. He continues as our Eastern Manager in our New York City office.

Our prime responsibility as publishers is to you, our readers. To the extent that you tell us what you like and don't like about *American Perfumer*, to just that extent can we tailor its content to your needs and expectations. We hope we hear from you often.

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Publisher

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Spice	DH-74
Para Odor	DH-122
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Cedar Wood	DH-132
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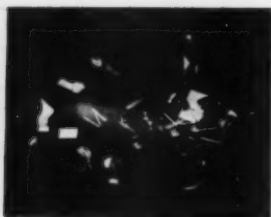
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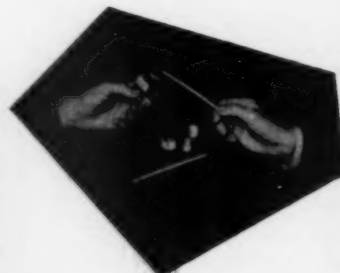
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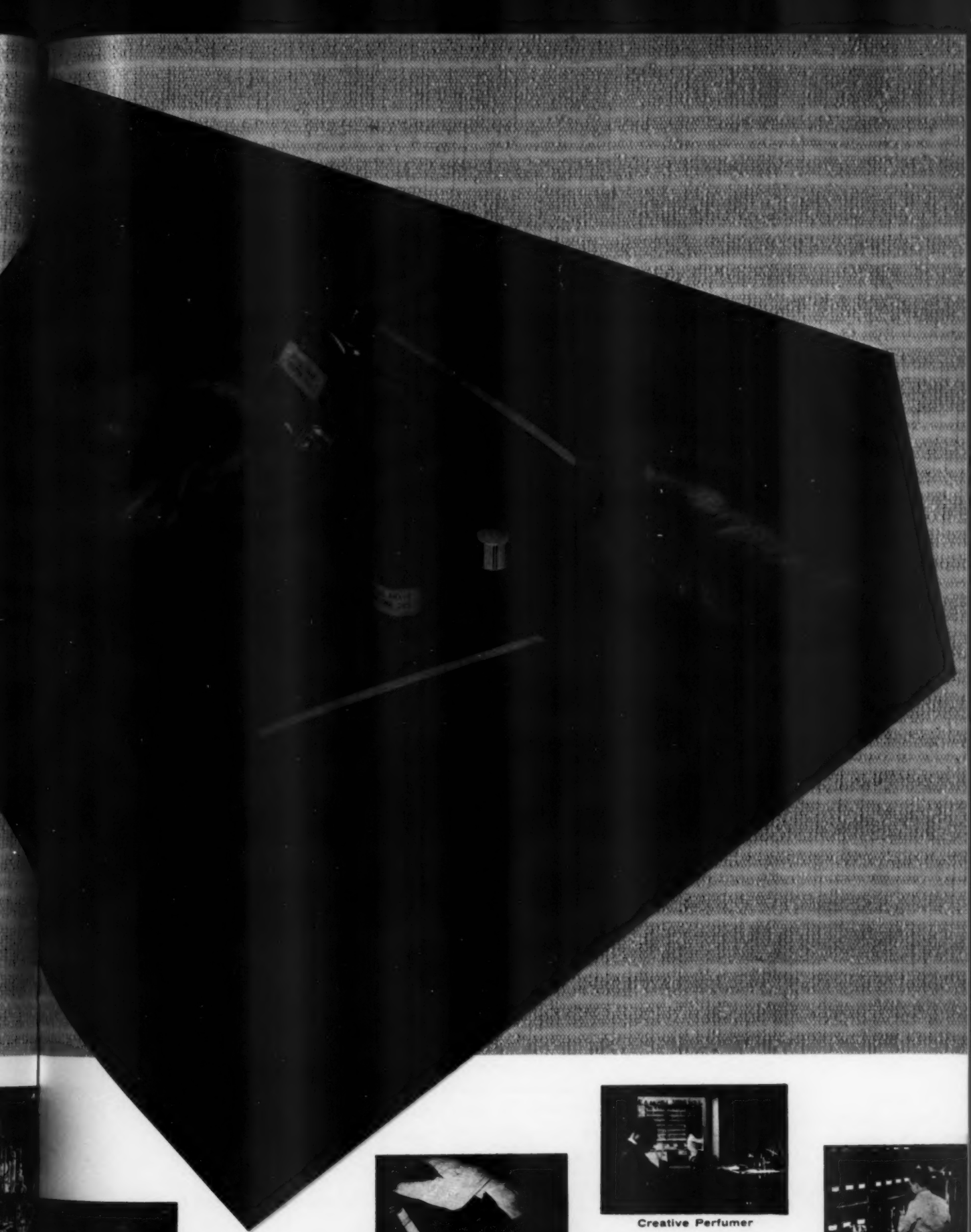
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MINUTE NEWS . . .

TGA Convention Committees Announced

The general chairman of the Toilet Goods Association 25th annual convention to be held June 26-30 in Poland Spring, Maine, will be John F. Hunsicker of *The Saturday Evening Post*. Other committees are as follows: Business Program, Charles F. Junod, Pacquin, Inc., chairman, Ivon H. Budd, Ungerer & Company, Hal Hutchins, *Redbook*, Bud Keeley, *Beauty Fashion*.

Entertainment, Eugene J. Moore, Richford Corporation, chairman, John M. Duncan, Continental Can Company, H. Robert Miller, White Metal Mfg. Co., James McInnes, Jr., Commercial Solvents Corp., Jack P. Jordan, Crown Cork & Seal Co., Richard Malmstrom, N. I. Malmstrom & Co. Golf Committee: Paul E. Forsman, C. H. Forsman Co., chairman, Michael Lemmermeyer, Aromatic Products, Inc., A. C. Burgund, Carr-Lowrey Glass Co., Donald Bush, Fritzsche Brothers, Inc., Alden Ludlow, Jr., U. S. Industrial Chemicals Co., Paul Bremer, Christian Dior Perfumes Corp.

Hospitality: Lamson M. Scovill, Scovill Mfg. Company, chairman, Dallas D. Roush, Owens-Illinois Glass Co., Walter Morton, Hazel-Atlas Glass Co., Robert C. Ring, The Hewitt Soap Co., Lee Simmons, Imco Container Corp., J. William Voit, George Lueders & Co. Ladies' Entertainment: Mrs. Alec J. Dedrick, chairman, Kay Colton, Morningstar-Paisley, Inc., R. Jane Williams, Schimmel & Co., Mrs. Jack P. Jordan, Mrs. Edward P. Morrish, Mrs. Russell Rooks, Mrs. H. B. Valerius and Mrs. Lee Simmons.

Helene Curtis to increase dividends

The directors of Helene Curtis Industries, Inc. voted the regular quarterly dividend of 10¢ per share plus 1% in stock on Class A shares payable March 15, 1960 to shareholders of record March 1, 1960.

Because of the increased company earnings and its strong cash position, the directors have declared their intention to increase the quarterly dividend to 17½¢ per share on Class A shares for the next quarterly payment payable on June 15, 1960 to shareholders of record on June 1, 1960. This is in line with the company policy of paying not less than 50% of earnings in dividends.

Lanolin Plus Names Executive Vice President

David W. Black has been named executive vice president of Lanolin Plus, Inc. Mr. Black most recently served as president and director of the American Losse Leaf Corp. Prior to this he was vice president of the Richard Hudnut Division of Warner-Hudnut Pharmaceutical Company.

1959 Sales Figures for Soap and Synthetic Detergent

A 1959 sales census conducted by the Association of American Soap & Glycerine Producers, Inc. has shown sales of soap and synthetic detergents up 4.4% in pounds and 5.1% in dollars compared to 1958 figures. Liquid synthetic detergents continued their sharp sales expansion and showed a 31.4% increase in pounds and a 25.9% increase in dollars.

Sixty-two manufacturers surveyed reported total sales of 4,269,877,000 pounds valued at \$1,093,450,000. Of this total 1,066,440,000 pounds and \$319,220,000 were attributed to soap and the remainder divided between solid synthetic detergents and liquid detergents.

The program of the Sixth Annual Open Symposium of The American Society of Perfumers has been announced. The title of the Symposium is "Abstract and Practical Thoughts in Perfumery". Fred C. Theile, Donald Dean and Robert Suffis of Shulton, Inc. will discuss "A Modern Approach to the Evaluation of Bergamot Oil." E. R. Van Liew of Givaudan-Delawanna, Inc., will speak on "Fragrance and the American Male." Alfred H. Moeller of Noville Essential Oil has as his topic "The Influence of Human Temperament in Odor Selection." Dr. Oliver L. Marton, Shulton, Inc., will discuss "Pitfalls in Perfumery" and Thomas Cifelli, Jr. of Richards and Cifelli will speak on "Perfumers and Patents." "The Abstract Concept in Perfumery" will be covered by Walter Lengsfelder of Fleuroma.

The Symposium will be held on April 28th at 2:00 P.M. at the Essex House Hotel, 160 Central Park South, New York City.

Farewell to Pierre Chauvet

"The soul's joy lies in doing"—Shelley

The French Perfumery Industry, that great family, is in mourning. Pierre Chauvet is no longer with us.

From the first symptoms of his illness, we realized that there was little hope, but the courage of Madame Chauvet and his daughter, and the care with which those close to him disguised the true nature of things, helped him to retain the illusion of an eventual cure.

Towards the end, when he suffered greatly, strong doses of analgesics not only gave him body relief but gradually lulled his consciousness. His perception of the world around him became clouded; logical reasoning processes, happy memories, and lively images of the past and bright processes of the future, all this life of the spirit was forthwith extinguished. Such was the will of God.

Pierre Chauvet, it was late in my own life that I first met you. We were friends, and I did all I could to get to know you better. I want to bear witness to the man that you were, without vain flattery, for only the truth is necessary to reveal your essential humanity.

Physically, you had the square-shouldered appearance of a wrestler. Your rugged face made me think in your youth you had been a practitioner of the noble art, but you assured me that this was not so.

Your whole personality announced that you were a man of action. You were always ready to give battle to men, events and adversity, in order to create a field of flowers, to construct a factory, to start work on a better product, and also to defend your ideas, to convince others and to take the lead. It was in this incessant struggle that you tasted the joy of living.

Perhaps you made yours the device of Marshall Lyautey, who was inspired by Shelley's line: "The soul's joy lies in doing."

If action was the essential mark of your life, the work you left behind shows how fruitful that action was. The scope of your work was astonishing, and also the rapidity with which it was realized.

It was in the South of France that you enlarged the Seillans factory, bringing its equipment up to date and instilling in all those connected with it, your own faith in the success by continuous effort.

It was your plantation of lavender and lavandin, and your distilleries in the Basses-Alpes, that little country so dear to your heart, of which you speak with emotion. There you were born and there, above all, you were able to relax. The landscape, the picturesque villages, the people and their customs, all of these spoke to you of your youth and of your childhood. Each return to them was for you a festival of the heart and spirit.

It was nevertheless and above all your work in Morocco, work of exceptional amplitude, to which even up to the last moment you consecrated the best of your activity.

You were proud of it, and I understand your pride.

A dozen domains, probably more than 1400 hectares

under cultivation: Rose, Jasmin, Tuberose, Orange Flower, Geranium; four judiciously located factories: such is the balance sheet.

Together we visited all these estates, whose names sounded a fanfare. When you spoke them, as though celebrating so many victories: Khemisset, Tiflet, Rabat, Port Lyautey, Tedders, Maaziz, Oulmes, Menerlaz . . .

Oddly enough, I call to mind Maaziz, at the time of the harvesting of roses. There were 110 hectares under a single tenant. The bowl shaped ground allows one to take in at a glance the entire plantation. From the distance, the women flower-gatherers advanced toward us in a long line. Behind them was nothing but green vegetation, where as in front of them broke a colourful wave of rose trees heavy in flower.

Such a spectacle left me silent. What could I say to you to convey my surprise, my sense of wonder. I simply shook you by the hand.

It remains for me to say something about the warm hearted man that you were. A first approach to you was not easy. You were distinguished by a certain reticence, even by a touch of scepticism. Your friendship was not given lightly, for you preferred to take time for reflection. I recognized, in our contacts, this deep-rooted feeling. But I can truly say that your friendship, once given, remained, that it was vigilant, eager to be of service, to sympathize with and to understand.

Of all memories we shared in Morocco, I would like to single out one of many, that raised you in my own esteem. It was in the year that preceded the accession of Morocco to independence, a year marked for the French by grievous trials. All your work seemed to be threatened. The plantations, factories, and even the security of the French people who surrounded you. Realizing the gravity of the situation to be taken you turned to me and aptly used the well-known phrase: "Je maintiendrai."

And in the turmoil, the exploitation of your estates continued; the harvesting of flowers was assured; the factories went on producing, in a perfect accord between French and Moroccans.

This must be, Pierre Chauvet the Moroccan, your greatest cause of pride, to have created a venture in which the social climate remained at "set fair", while Morocco acceded to independence.

Pierre Chauvet, rest in peace

Al Seillans, in the Basses-Alpes, in Morocco, your work remains. It is a duty for all those who have been associated with you to insure its continuation. You know that they will not fail, because you yourself knew how to choose them.

Thus, and in the most noble fashion, they keep and honour your memory.

E. P. Meunier

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Questions and Answers

O. What is the function of ethyl stearate often included in baby oil formulations? In such formulations is ethyl stearate absolutely safe, that is, nontoxic, nonirritating and nonallergic? Could you advise who manufactures this ester? F. R., Spain

A. The use of ethyl stearate in cosmetics is covered by a U. S. Patent and possibly patents in other countries. We would suspect that it would have properties similar to those demonstrated by isopropyl palmitate-myristate mixtures which are commonly used. Ethyl stearate should be comparatively nontoxic if properly made but you would have to test the finished product to be certain. The product is sold by the R. W. Greeff and Co., Inc., 10 Rockefeller Plaza, New York 20, New York, who also have agents in Europe.

Q. We would appreciate receiving more details on the following items as mentioned in "Desiderata."

The new new quat germicide, diphenyl mercuric ammonium propionate

The antibacterial cloth and extruded plastic sheeting

The dextro rotary lactic acid

We enjoy your feature except for the exasperation of writing for sources before we can take further action. I guess this is your method of control over "reader interest." Do you have any technical data on desoxyribonucleic acid? Who makes the oleyl oleate mentioned some time ago? C. L. S., New York

A. The mercurial quaternary germicide is available from the Metalists Corp., 200 Wagaraw Rd., Hawthorne, New Jersey. The dextro rotary lactic acid is obtainable from the M. R. Petrasch and Co., Dornbirn, Austria. "Eversan" is the name of an organic zinc complex developed by Ions Exchange and Chemical Corporation, 48 Leonard Street, New York 13, for application to cloth to make it

antibacterial. The antibacterial plastic sheet is made by the Compo Division of Chicago Molded Products, 1020 Kolmar Avenue, Chicago, Illinois. We are sorry you find it frustrating to write in for sources of supply, but it is our only way of knowing what sort of things are of interest to our readers. This in turn then guides editorial policy. We do not have any data to send you on desoxyribonucleic acid. The oleyl oleate mentioned some time ago is made by the Deutsche Hydrierwerke, A. G. (Dehydag), Dusseldorf, Germany. They have distributors in the principal U. S. cities.

Q. We have been looking for an antioxidant known as "Antoxan A" which is supposed to be made in this country. We have been unable to find it anywhere and cannot even find it listed in the INTERNATIONAL ENCYCLOPEDIA OF COSMETIC MATERIAL TRADE NAMES. Any information you may have on this product will be gratefully appreciated. L. J. C., Maine

A. The antioxidant "Antoxan A" is unknown to us too. We do have a listing of the two products which are close to the one you mentioned. Perhaps one of these companies has made another antioxidant which is identical to the one that interests you.

Antox. Antioxidant. E. I. duPont de Nemours Co. Wilmington 99, Delaware

Antox. Antioxidant. Titan Products, Inc. Mill Road and Wayne Street Jersey City, New Jersey

This is scanty information, but it is the best we can do at this time.

From time to time suggestions have been and will be made in this magazine with respect to processes, devices, materials, appliances, equipment and the like. It is not practicable for the writers and editors to have a patent search or examination made in connection with each such suggestion. Our readers are, therefore, requested and indeed urged to determine for themselves whether any patent or other right will be violated before acting on any such suggestion.

AMERICAN AROMATICS

- Perfume Compositions

- Essential Oils

- Aromatic Chemicals

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SCENTS



PFW

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AMERSFOORT, HOLLAND • PARIS, FRANCE • BREMEN, GERMANY • BRUSSELS, BELGIUM • SOFLOR LTD., PERIVALE, ENGLAND

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Maison G. deNavarre, M.S., F.A.I.C.



Aptitude Tests

Not being modern in any respect on formal tests, I have never been a fan of the I.Q. and related tests. This influenced my coolness to the use of aptitude tests particularly by employers. My direct experience with them ended up with the individual so tested and approved to fail miserably when hired. They can determine ability when properly worked out but they won't predict a worker's success at a job. Yet it is the latter with which employers must be most concerned.

A recent book "10,000 CAREERS" published by Wiley is a study of four years of aptitude testing of 10,000 World War II airforce men both in service and later in their careers. The conclusion here too is that the aptitude test can determine ability but not success at a career so chosen.

Carcinogens

A statement appearing in the J. INVEST. DERMATOLOGY 34, 58, 1960, states as follows: "...for light skinned individuals the psoralens are possibly carcinogenic and we do not advocate them except where definitely indicated in selected cases of vitiligo."

How can a doctor justify the use of a possible carcinogen in any treatment? Certainly vitiligo would be my choice in preference to cancer. I am certain he too would prefer it.

In the light of a possible "cancer

clause" in new food and drug legislation, why shouldn't this extend to medication by a doctor? Cancer is cancer. If one provokes it knowingly without being held to account, something is wrong somewhere.

Mechanism of Action

Speaking at the Society of Cosmetic Chemists' Seminar last September, Blank made the statement "... by what mechanism a given cosmetic may produce the desired results."

This is the goal, not only of the cosmetic scientist, but the pharmacologist, medical practitioner, chemist, physicist and all other scientists—"by what mechanism." Our knowledge of mechanisms is continually increasing. Whether we shall ever know all, only time can tell.

It seems to me that man has made use of many discoveries before he knew the mechanism by which his discovery operated. If we waited until we knew the mechanism by which drugs produced their results, we would still be waiting for some if not all of our simple remedies. Furthermore, we theorize on a mechanism by which something produces its results, only to find in the light of later knowledge that our earlier theory may have been quite wrong.

We should, and we do, strive to know how a cosmetic product works. We would know more about

it perhaps if a segment of scientifically trained men had dogmatically not said that the skin was impermeable—that things applied to it were as water on a duck's back and hence wasted. With such prejudice our knowledge of "mechanism of action" had to wait for a later day of greater understanding. It will not be too long before we know the how, the when, where and why of cosmetic applications to the human skin, if we have open minds on the matter.

Brushless Shave Cream

Prices of Bromborough (England) have just issued a new booklet (#5) on the use of fatty acids and alcohols in toiletries. One of the interesting formulas tells you how to make a good brushless shave cream.

Stearic acid	19.0%
Propylene glycol	10.0%
Potassium hydroxide	1.0%
Cetyl alcohol	0.5%
Mineral oil	2.0%
Isopropyl myristate	1.0%
Water	66.5%
Perfume and preservative q.s.	

The stearic acid used contains 52% palmitic, 42.5% stearic, oleic 4.5% and myristic acid 1.0%.

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Russia and Patents

According to an editorial in the MANUFACTURING CHEMIST, it is possible for a foreigner to get a patent in Russia. The law of April, 1959 is similar to patent laws of other countries. However, much like the system in the U.S.A. prior art is determined by search. The invention must have utility. Renewal fees are paid annually for the life of the patent namely 15 years. Infringement action can be brought before the courts. Of course there is a lot more to it than these few words but that is the meat of the matter.

Honors

A letter dated February 15, 1960 from President Robert H. Marriott of the British Society of Cosmetic Chemists reads in part as follows:

"... At a Special General Meeting of the British Society it was proposed that you be elected an Honorary Member of the Society of Cosmetic Chemists of Great Britain. . . . the resolution was passed unanimously.

"It is my pleasant duty to write to you to inform you of this resolution and at the same time, to say how pleased everyone was to have the opportunity of electing you as the third Honorary Member of our Society."

To which I replied, "I am honored and delighted. . . ." It is my hope to accept the parchment in person in London at the Annual General Meeting on May 27th, at which time I can also renew my

acquaintance with my British contemporaries in the Society.

NOTES

Finely divided aluminas of the alpha and gamma types are being offered by the J. T. Baker Chemical Company, for possible use in dentifrices among other products Some interesting observations on the utility of linear phosphates and polyphosphates in foods mention that the cyclic metaphosphates are apparently not altered by the body to the useful orthophosphates. These data are found in the Quarterly Bulletin of Food and Drug officials, January, 1960, p. 47 Coming out of Italy is a product entitled "Atomic Cream" purported to contain placenta extract, hormones and vitamins, and "activating alpha rays" in some manner A new drug on the German market contains 10 mg. of 5-phenyl-2-imino-4-oxo-oxazolidine in tablet form to relieve fatigue, exhaustion and anxiety. It is said to be mild but sure Work reported by Fischer in Zurich indicates that salves containing 0.05 to 0.1% colchicine have a cytostatic effect on basal cell carcinoma of the skin Strehler thinks that ageing is due to clinkers formation in body cells, said clinkers being a lipofuscin age pigment according to C & EN for January 18, 1960, p. 40 Congratulations to our British contemporary, the CHEMIST AND DRUGGIST who have celebrated their centenary last November. Long life and continued progress to them B-propiolactone vapor at the rate of 1 mg./liter of

air for 15 minutes, inactivates the viruses causing smallpox, yellow fever, psittacosis, and the rickettsia *coxiella burnetii* according to workers at the U. S. Army Chemical Corps at Fort Detrick (App. MICROBIOLOGY, 8, 39, [1960]) Butler's "Queen Substance" has been isolated and characterized to be related to 10 hydroxy- Δ^2 -decanoic acid found in royal jelly. It has a molecular weight of around 190 (NATURE, 184, #4702, 1871, [1960]) Mills finds that a coal extract is bacteriostatic toward both gram-positive and negative organisms Jean Crobolona's observations at the Congress on essential oils last year at Grasse indicate that old orris root has a higher irone content than fresher root. Maybe that is why orris powder made a good sachet base Dow has developed a magnesium hydroxide containing 60% mg. (OH)₂ which is still fluid It's the 60th anniversary of aspirin. It got its name because salicylates have been found in Spirea. Salicylic acid had been also called Spirin. A-spirin was the name given to the acetyl compound according to our British contemporary CHEMICAL PRODUCTS Wichterle and Lim suggest a glycol methacrylate polymer as a hydrophylic gel for biological uses Soap increases the permeability of the barrier layer of the skin but does not destroy it as do some solvents (NATURE, 185, 18, [1960]) A German supplier offers Carotene Ricinate for hair and scalp lotions to be used in 2-10% concentrations.



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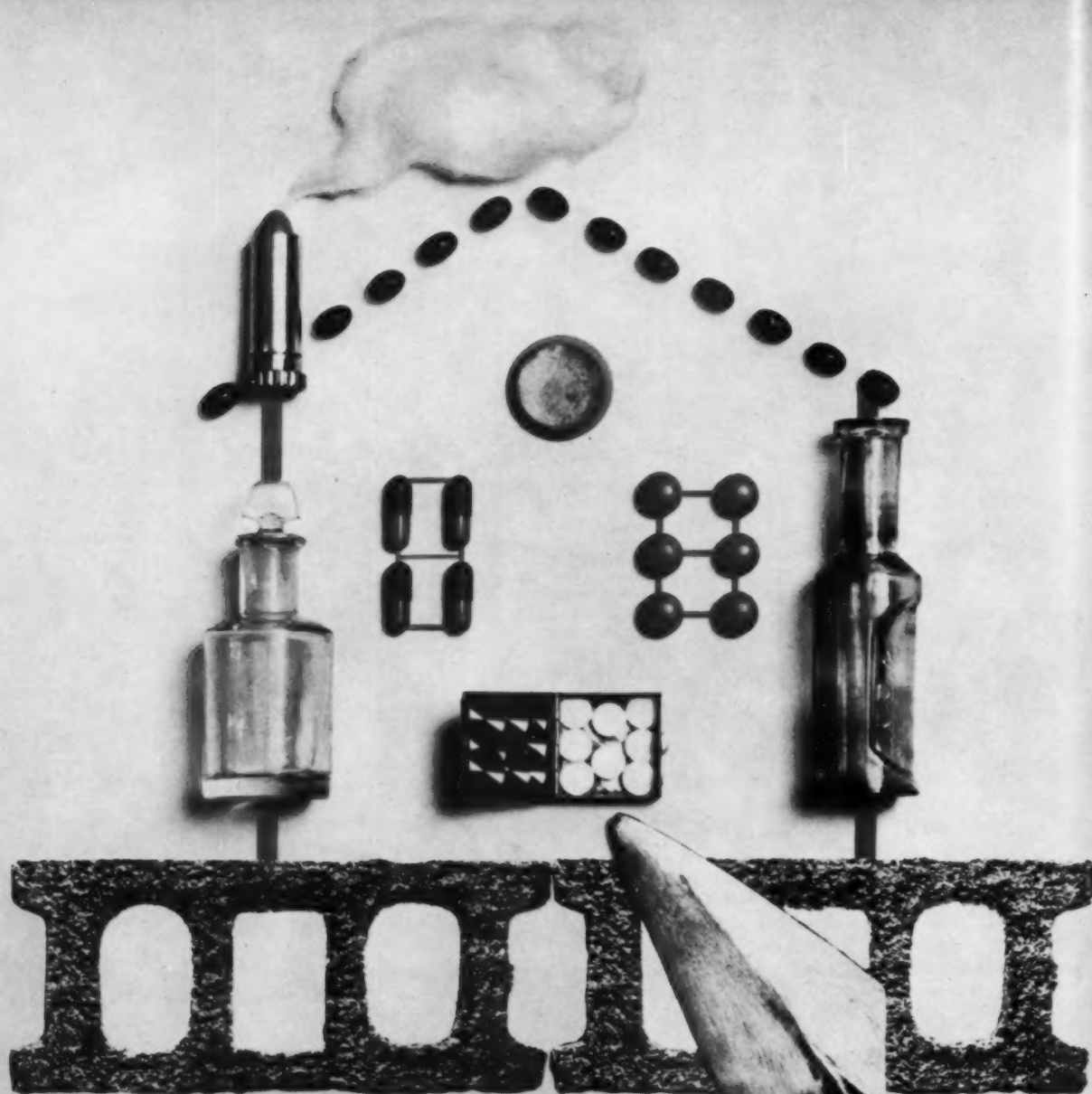
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The scope of fatty acids and derivatives in cosmetic formulations

BY GABRIEL BARNETT

COSMETIC RAW MATERIALS derived from animal and vegetable fats, oils, and waxes constitute an ever-increasing portion of modern cosmetic formulations. The great variety and novelty of present-day cosmetics has been made possible only because of the ready availability of these naturally-occurring esters, and the ingenuity of the chemical industry utilizing highly specialized techniques such as:

1. Fractional distillation
2. Solvent crystallization
3. Ozonolysis
4. Hydrogenation
5. Oxidation
6. Ethoxylation
7. Molecular distillation

Completely new approaches to cosmetic formulation have resulted from the abundant flow of fat-derived chemicals, which in turn have been used for further synthesis of newer raw materials.

Practically every cosmetic and toiletry product—cream, lotion, powder, lipstick, make-up, shampoo, dentifrice, hair dressing, creme rinse, cologne and deodorant sticks, shaving cream, antiperspirant, para hair dyes—has one or more components which were derived from a naturally-occurring fat, oil, or wax. The almost complete dependence of most cosmetics on these raw materials becomes strikingly evident when one attempts to list those products not containing fat-derived products.

Fat derived materials are equally varied in their properties and functions. They serve as emollients, thickeners, plasticizers, solvents, emulsifiers, wetting agents, dispersing agents, suspending agents, detergents, and solubilizers. Many surface-active agents have emollient properties and are most versatile in their wide range of hydrophilic-lipophilic characteristics. The relative occlusive or barrier effect of oil and wax films on the skin may be modified by the use of the various surface-active agents.

These naturally-occurring fats and oils offer one of the largest potential sources of raw materials for the preparation of chemical synthetics. The cosmetic materials derived from fats and oils are given below according to their chemical types, together with im-

portant examples for each classification:

1. Triglyceride esters—cattor, sesame, peanut, safflower, soybean, coconut oils and tallow.
2. Fatty acids—lauric, palmitic, myristic, stearic, oleic, linoleic, ricinoleic; hydroxystearic, arachidic, behenic, and arachidonic acids.
3. Fatty alcohols—lauryl, cetyl, stearyl, oleyl, talow, ricinoleyl, linoleyl, and lanolin alcohols.
4. Soaps—sodium, potassium, ammonium, mono-, di-, and triethanolamine, mono-, di-, and triisopropylamine, and amino glycol salts of fatty acids.
5. Detergents—
 - a. Alkyl sulfates from coconut oil fatty acids
 - b. Amide sulfonates
 - c. Ester sulfonates
 - d. N-acyl sarcosinates
 - e. Alkylolamides
 - f. Amines
 - g. Alkyl beta-amino propionates
6. Cationic antiseptics—
 - a. Quaternary ammonium compounds
 - b. Morpholinium compounds
 - c. Pyridinium compounds
8. Polyhydric alcohol esters—propylene glycol, glyceryl, sorbitol, and sorbitan fatty acid esters.
9. Ethoxylated fatty acids—polyethylene glycol mono- and di-fatty acid esters.
10. Ethoxylated fatty alcohols—polyethylene glycol ethers of cetyl, stearyl, oleyl, and lanolin alcohols.
11. Ethoxylated sorbitan esters—Tweens
12. Branched-chain high molecular weight alkyl esters—hexadecyl myristate

$$\begin{array}{c} \text{C}_6\text{H}_{13}-\text{CH}-\text{CH}_2-\text{COO C}_{13}\text{H}_{27} \\ | \\ \text{C}_8\text{H}_{17} \end{array}$$
13. Lanolin derived fatty acids and fatty alcohols—

Gabriel Barnett is research chemist for Coty, Inc. This paper was presented at the Fatty Acid Symposium of the 33rd Annual Convention of the Association of the American Soap and Glycerine Producers, Inc., January 20, 1960.

this wax ester yields normal fatty acids (even-numbered C₁₀-C₂₆), iso fatty acids (even-numbered C₁₀-C₂₈), anteiso fatty acids (odd numbered C₉-C₃₁), and hydroxy fatty acids (even-numbered C₁₂-C₁₈); and fatty alcohols (aliphatic, sterol, and "triterpenoid").

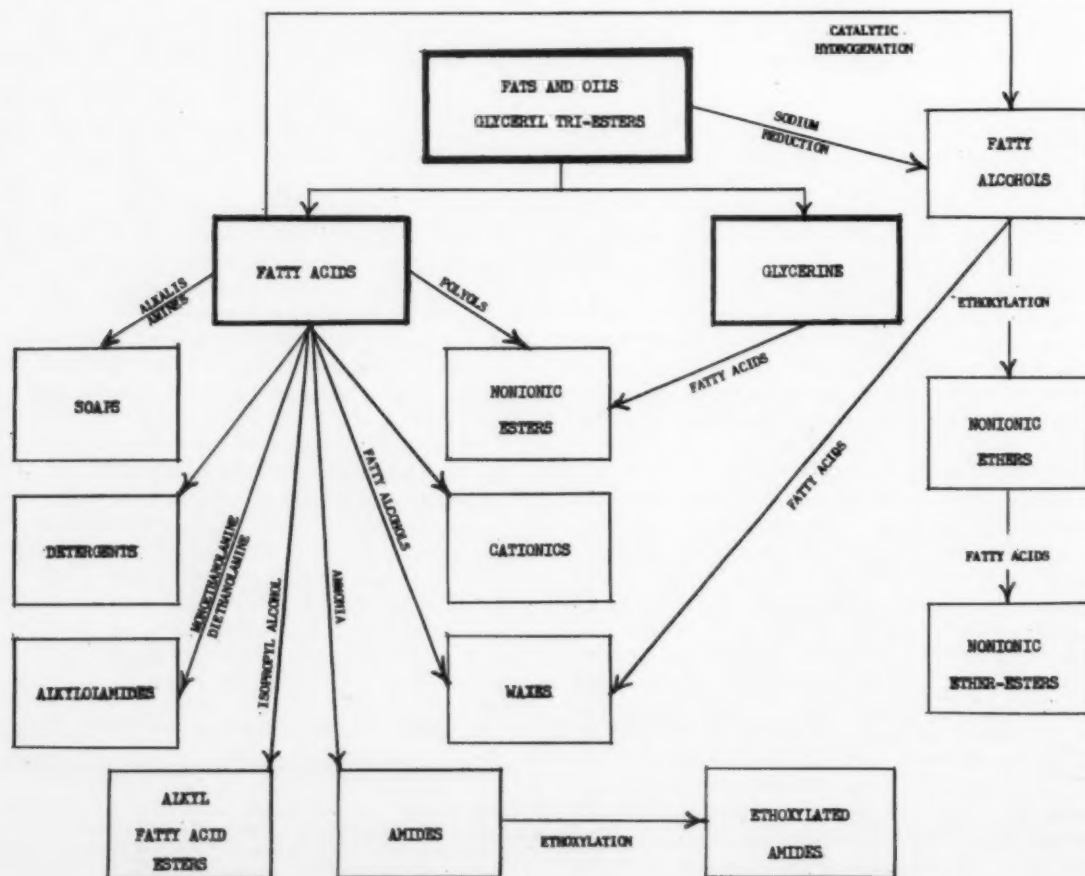
14. Lanolin derivatives—lanolin fatty acids to form amine soaps; lanolin fatty alcohol mono- and poly-esters of ricinoleic and linoleic acids; acetate esters; ethoxylated ethers.

The availability of the many fatty acids listed previously and the ever-increasing variety of chemical derivatives has given rise to serious problems regarding chemical and physical specifications. There are perhaps ten companies supplying fatty acids to the cosmetic industry and to specialty manufacturers of fatty acid derivatives. There are at least a dozen companies in the latter category, each with their own special manufacturing procedures, supplying fatty acid derivatives to the cosmetic industry. It is therefore not surprising that this state of affairs often results in a given product which differs significantly when compared to another source of the same material. As a result a direct substitution of one source for another is not possible and reformulation becomes necessary.

A discussion of the chemistry of glyceryl monostearate illustrates the differences one finds among the many suppliers. This extensively used emulsifier is generally made from triple-pressed stearic acid,

consisting mostly of palmitic and stearic acids with small amounts of myristic and oleic acids. In addition to percentage differences of these four fatty acids from different sources, there is another variable in regard to its method of manufacture. The term "triple-pressed" now includes products prepared by solvent crystallization, vacuum distillation, and pressing in combination with either procedure. The different crystalline types obtained are a function of processing techniques, and the effects of these forms are of considerable significance in cream consistency and fracture (1). These chemical and physical differences carry over into the preparation of glyceryl monostearate and other fatty acid esters.

The case of the so-called "Glyceryl Monostearate" is further complicated because it consists of mono-, di-, and tri-esters of palmitic, stearic, oleic, and myristic acids, as well as 1.5% free fatty acids and about 3.5% glycerin. Hence, with four fatty acids in "triple-pressed" stearic and with three types of esters possible, there are 12 molecular species present in the mixture. The four major components are glyceryl mono- and di-palmitate, and mono- and di-stearate, with small amounts of oleate and myristate esters (total mono-ester and di-ester average about 45-47% each, and about 1-5% tri-ester). And since there is more palmitic than stearic in "triple-pressed" stearic acid (according to the 55/45 ratio), there is more mono- and di-palmitate present than stearate ester. So it would seem that what the industry sells as "glyceryl



monostearate" is actually a misnomer. A more striking example is the so-called "ethylene glycol monostearate" which contains 50-60% di-ester and 43% mono-ester. While the acid number, saponification value, and melting point of alternate samples may meet specifications, the amounts of the 12 esters (in glyceryl monostearate) may vary sufficiently to change emulsification behavior and product stability. Another critical factor is the nature of the crystalline forms (polymorphism) of glycerides observed in different samples (2), and its significance in creams and lotions (3).

It is therefore strongly recommended that suppliers of these fatty acid esters include in all shipments the complete specifications for these variables. This should include the exact chemical composition of the fatty acids, its method of manufacture, the mono-, di-, and tri-ester content, free fatty acid, and the other standard data such as melting point, color, iodine and saponification value.

The same recommendation is suggested to manufacturers of the other fatty acid derivatives described earlier. The importance of the exact fatty acid or fatty alcohol composition is often overlooked in detergents, alkyl esters, and alkyl ethers, with major emphasis generally placed on its functional properties and elementary specifications. A detailed and complete physical and chemical analysis of the components used in its manufacture as well as of the finished material would be of considerable value to the cosmetic industry, where uniformity and stability of products is of prime importance. On those rare occasions when a production lot shows a loss of emulsion stability, a complete analytical record of the raw materials is invaluable in determining if the cause is chemical in nature.

There is one area in the fatty acid derivative field which has not been exploited, and is offered for consideration as one which may have potential market value, particularly in the cosmetic industry. Now that single fatty acids of extremely high purity are available, the manufacture of these glyceryl and other polyol esters would have decided advantages over the conventional fatty acid ester mixtures. For example, glyceryl monostearate made with 93 or 97 percent stearic acid would avoid the inevitable differences in triple-pressed stearic acid composition, and perhaps might become the standard for this material. Glyceryl monopalmitate made with 93% palmitic acid is now possible, and which may be preferable to other glyceryl esters in certain instances. In effect these high purity fatty acids would permit a broader range of hydrophilic-lipophilic characteristics in the glyceryl fatty acid ester emulsifier family, thus adding greater versatility to their use.

An even greater refinement in approaching true chemical purity of nonionic esters is the molecularly distilled glyceryl monostearate containing 90-95% mono-ester. The application of molecular distillation in the preparation of these esters using high purity fatty acids and various polyols would be the ultimate in supplying the cosmetic industry with the most uniform and nearly chemically pure fatty acid esters. Here is one area which has hardly been explored, probably because of the cost factor, but certainly merits additional research to create a larger market

with its attendant economies in production.

There is another area in fatty acid technology which would command more than academic interest by the cosmetic industry. This involves a study of the properties of fatty acids with more than 18 carbon atoms, such as arachidic (C-20), behenic (C-22), and lignoceric (C-24) in the saturated series; and arachidonic (C-20 tetra-unsaturated) and clupanodonic (C-22 penta-unsaturated) as members of the important essential poly-unsaturated fatty acids. The availability of these unusual fatty acids, free from color, odor, and impurities, would certainly strike the imagination of cosmetic research chemists, and perhaps give rise to a new series of derivatives with valuable properties not found in conventional types.

One aspect of fatty acid manufacture which permits greater flexibility in formulation is the availability of very high purity fatty acids. Lauric acid is available with 95% C-12 content, myristic acid with 94% C-14, palmitic acid with 93% C-16, and stearic acid with 94-97% C-18.

The definite crystalline structure of the conventional 55:45 palmitic/stearic ratio is well known, which may be contrasted with a non-crystalline structure at a 40:60 ratio. The relative crystalline structure of a fatty acid mixture is of critical importance in shaving and vanishing creams, in regard to consistency, rheology, fracture, pearliness, and product stability. By custom blending the single high purity fatty acids it may be possible to achieve the desired degree of crystallinity or amorphous nature.

In soap shampoo formulations the foaming properties, bubble size, foam stability, solubility, and detergent action are a function of the fatty acid composition. These properties may be controlled to a degree which was once impossible before the availability of these single high purity fatty acids.

Shampoos and dentrifices which use synthetic detergents contain a fatty acid component which is part of the surface-active agent, generally derived from coconut oil.

The fatty acids are used in high concentrations in shaving and vanishing creams, soap shampoos, permanent hair coloring, and cologne and deodorant sticks; and in low concentrations in hand lotions, cleansing and emollient creams and lotions, and liquid make-up.

The nonionic fatty acid esters and fatty alcohol ethers serve as emulsifiers, emollients, thickeners, and solubilizers, and are used in these same creams, lotions, and make-up, including lipstick, face powder, mascara, and eye shadow.

The complete dependence of almost all cosmetic formulations on natural fats and oils and fatty acid derivatives is summarized in the drawing. One could list a long series of cosmetic formulations, each containing one or more of these derivatives, but they are readily found in cosmetic books such as "Cosmetics: Science and Technology" (4), and suppliers' brochures.

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Decomposition of medicaments due to excipients and containers, and its prevention

BY T. D. WHITTET

The choice of the best excipients for the formulation of remedies is of vital importance in ensuring the stability and efficiency of the resulting preparations.

It is therefore essential that pharmacists should have as complete a knowledge as possible about all the chemical, physical and pharmacological properties used for the formulation of medicaments, including vehicles and excipients as well as those of the active ingredients themselves. Although in Great Britain the term excipient is usually restricted to substances used in the formulation of tablets, capsules and pills; in this paper it has been taken to include any substance used in the formulation of a medicament.

Ideally one would not use as an excipient anything likely to cause decomposition of the medicament, but it is not always easy to ascertain whether any such effect is likely to occur. The field is so vast that it is not possible, in one paper, to cover more than some selected parts of it, but I have endeavoured to make the examples discussed deal with as wide a range of preparations as possible and have included as many personal observations as I can recollect.

I have also, in a few instances, taken a wide interpretation of the term "decomposition" and have included factors causing reduced efficacy or increased toxicity of medicaments which may not be due strictly to chemical decomposition. I have taken the view that anything which interferes with the effectiveness or increases the danger of a medicine is as serious as actual decomposition of the active ingredient.

Since faulty packing or storage of medicines can invalidate the best formulated products, it is appropriate that the effect of containers on remedies should be discussed in the same paper.

As the earlier speakers in this symposium have dealt with the beneficial action of preservatives, antioxidants, solubilising and suspending agents, etc., I shall confine my paper to examples where such sub-

stances have given unexpected and deleterious results.

To make the paper as precise as possible I have dealt first with the incompatibilities and factors causing instability of several important groups of drugs and with those of excipients used in many types of preparations. Subsequently examples of unexpected results of formulations are discussed under the heading of the particular type of pharmaceutical preparation. A very extensive list of references is appended to the paper, since it is impossible to do more than mention some of the papers cited and the inclusion of all the references is therefore essential.

GENERAL INCOMPATIBILITIES OF SOME IMPORTANT GROUPS OF DRUGS ADRENALINE AND RELATED PRODUCTS

Adrenaline and related products are subject to direct oxidation and to a secondary indirect chain reaction which can occur in the absence of free oxygen. Decomposition is accelerated by metallic ions, especially ferric, ferrous, cupric and chromic ions. Metabisulphite alone will not prevent this type of decomposition and it seems advisable to add both an antioxidant and a chelating agent to solutions of these compounds. The rate of decomposition increases with rising pH and in alkaline solution the presence of metabisulphite may even accelerate the secondary reaction. It is obvious that solutions of salts of adrenaline and related compounds must be protected from oxygen and stored in containers which do not liberate alkali or metallic ions.

ANTIBIOTICS

With such a diverse and biologically active group

This paper is by T. D. Whittet, S.Sc., Ph.D., F.P.S., F.R.I.C., D.B.S. It is a shortened version of a paper read at the Symposium on Drug Stability at the 19th International Congress of Pharmaceutical Sciences, Zurich, in September, 1959.

of substances as the antibiotics it is not surprising that many incompatibilities have been reported. Many of these are listed in the full paper and attention is drawn to the following important examples.

Bacitracin is incompatible with many common ointment bases and care must be taken to choose one which does not cause inactivation. Neomycin is incompatible with anionic substances and with some metallic ions. It is very unstable in ointments containing hydrous lanolin.

Penicillin has so many incompatibilities that a good rule is not to formulate it with any excipient not known to be compatible, without carrying out activity tests.

Although streptomycin solutions darken on storage this does not indicate loss of activity and we have found that plain unpreserved solutions cause considerably less pain on injection than preserved ones.

The tetracyclines in solution are very susceptible to changes of pH. Metallic ions may affect both stability and absorption.

VITAMINS

Many of the vitamins have narrow ranges of pH for stability, e.g. aneurine, folic acid, cyanocobalamin and ascorbic acid. Several are damaged by oxidation, e.g. aneurine, nicotinic acid, riboflavine, p-aminobenzoic acid, folic acid, cyanocobalamin, ascorbic acid, vitamins A and D. Various metallic ions cause decomposition of the following: aneurine, riboflavine, folic acid and cyanocobalamin. Nicotinic acid is particularly liable to dissolve copper.

It should be remembered that the sulphite group of preservatives can cause decomposition of some vitamins such as aneurine and cyanocobalamin. When formulating multivitamin preparations, possible interaction between the various vitamins must be considered. Riboflavine, in particular, is liable to cause decomposition of other vitamins such as ascorbic and folic acids.

GENERAL INCOMPATIBILITIES OF VEHICLES AND EXCIPIENTS

Developments in the past few years have provided pharmacists with many new vehicles and excipients and these are being used for an ever-increasing range of medicaments, many of which are highly reactive substances. The possibilities of incompatibilities are therefore enormous and we must record and publish every example we can find.

Even with as simple a substance as water, difficulties may arise. Distilled water can contain oxygen, carbon dioxide, copper, lead and traces of other metallic ions. Deionised water is practically metal-free and has advantages for some preparations. Oils and ethyl oleate may contain peroxides which also may be a cause of decomposition when these are used for solvents.

Dyes may react with many medicaments and may inactivate certain ionic bactericides. Some dyes are bleached by sulphites.

The incompatibilities of the various preservatives are too numerous to mention here in detail, but many are listed in the full paper and the principal references are given. The instability of chlorbutol

towards heat and of thiomersal towards storage are mentioned.

As I pointed out at the Rome Congress in 1951, metabisulphites undergo considerable decomposition in air, especially on heating in solution. Other workers have confirmed this. The decomposition is accompanied by a rise in pH and this may accelerate the decomposition of some substances such as sodium aminosalicylate. Bamann and others have shown that metabisulphite can cause hydroxylation of various compounds with loss of activity.

The comparative instability of sodium metabisulphite is, of course, responsible for its protective action, since it is preferentially oxidised; but it seems that an appreciable portion may be lost during sterilisation before it has time to exert its antioxidant effect. It is probably advisable, therefore, to replace the air with nitrogen in ampoules containing oxidisable substances, even in the presence of metabisulphite. Whenever possible ampoules should replace rubber-capped bottles for such solutions.

Since sodium sulphite appears to be more stable than the metabisulphite and is an efficient antioxidant, it may be preferable to use the sulphite for pharmaceutical preparations.

Sodium formaldehyde sulphonylate, which is also used as an antioxidant, is of doubtful stability and can react with procaine hydrochloride to form a compound without analgesic activity.

THICKENING AND SUSPENDING AGENTS

The natural gums, such as tragacanth and acacia, contain enzymes which may spoil many preparations. Numerous new synthetic or semi-synthetic excipients are now available. These have advantages, but also many incompatibilities which are listed in the full paper. Extensive research in this field has been carried out by Higuchi and his collaborators.

Non-ionic surface active agents have been shown to inactivate many antiseptics and fungicides. An unusual effect of the efficiency of new emulsifying agents is the fact that in very fine emulsions oils are much more readily oxidised.

BUFFERS AND pH ADJUSTMENT

Adjustment to the optimum pH is becoming increasingly important in controlling the stability of drugs but it must not be forgotten that the optimum pH for stability may not be that for maximum pharmacological activity. In some cases adjustment to the correct pH with a strong acid may be preferable to the use of buffers.

FLAVOURING AGENTS AND PERFUMES

Care must be taken in the choice of flavouring agents, since, in addition to chemical incompatibilities, some flavouring agents may render certain drugs even more obnoxious. Some perfumes may react with drugs to produce unpleasant odours and others may be decomposed by metallic containers.

UNUSUAL EFFECTS WITH VARIOUS FORMS OF MEDICAMENTS

TABLETS AND CAPSULES

Little attention has been paid to the diluents used

for tablets and capsules, substances such as lactose, sucrose, dextrose and calcium phosphate merely being regarded as inert fillers. Evidence is accumulating, however, that, apart from actual incompatibilities, the filler may have a profound effect on the absorption as well as on the stability of the medicament. It has been shown that calcium and magnesium salts markedly lower the blood levels obtained after giving the tetracyclines orally. Calcium lactate and phosphate have been used as fillers for preparations of these drugs. My colleagues and I found that calcium phosphate caused an increase in the toxicity of calciferol. I also found incompatibilities of sugars, especially monosaccharides, and of metallic ions with aminophylline. It has been reported that the chocolate base formerly used for tablets of glyceryl trinitrate reduces its activity and that stearates and alkalis cause decomposition of aspirin.

Another important matter in the formulation of tablets is to ensure that the excipients used do not interfere with either the disintegration or the absorption of the medicament.

Sperandio et al. pointed out that the efficiency of a tablet is to a great extent influenced by the speed with which it disintegrates. They stressed the importance of distinguishing between disintegration, the mere breaking up into particles or granules, and solution. Parrott et al., in their investigation into drug release from solids, emphasised the point still further. They state "While the disintegration time of a tablet does influence the rate of drug release to the body, a more important aspect is the rate of release from the primary drug particle since solution of the drug is essential in order for absorption to take place. Thus, the release of a drug from the primary particle and its subsequent availability to the body is governed by the dissolution rate of the particles."

Evidence is accumulating that both the particle size of drugs and the type of binding agents may affect its absorption. It has been shown that, unless specially treated, lipophilic drugs may not disintegrate. The type of granulating agent has also been proved to affect the disintegration of sodium bicarbonate tablets.

Evidence, soon to be published from Imperial Chemical (Pharmaceuticals) Ltd., shows that the particle size affects the absorption of phenindione. The same effect occurs with the phenyl ester of para-aminosalicylic acid and with the corticosteroids. Some forms of enteric coating have been found to cause tablets to pass through the body unchanged.

INJECTIONS

Many factors influence the stability of injections. Some of these have already been mentioned and a few unusual reactions will now be discussed. As simple an ingredient as dextrose may cause several unexpected effects. Heating with dextrose has been reported to destroy several amino acids and vitamins. This may be of importance in the preparation of intravenous nutrient solutions. Sodium lactate has been reported as causing destruction of dextrose on heating. Dextrose also undergoes caramelisation on autoclaving with certain salts, especially in alkaline solution. I have found that this can be prevented by

metabisulphite without producing any undesirable effects in intravenous infusion solutions.

On autoclaving, local analgesics having a primary amino group, with dextrose, glycosides without analgesic are formed. Similarly, my colleagues and I have found, sodium para-aminohippurate forms a glycoside which gives a false result in renal function tests. Obviously, dextrose should not be used to adjust the tonicity or baricity of local analgesic solutions or for adjusting the tonicity of solutions of sodium para-aminohippurate. Procaine is subject to oxidation and this is accelerated by metallic ions which may be liberated from containers.

Sodium chloride may precipitate colloidal substances, especially dyes, from solution. Somers and I found this to occur with injection of Congo red with a marked increase in toxicity.

Some stabilisers may actually cause decomposition instead of preventing it. For example, calcium saccharate and edetic acid have been reported as accelerating the precipitation of calcium gluconate instead of preventing it. It has also been reported that sealing of solutions of sodium aminosalicylate with carbon dioxide accelerated the rate of decarboxylation instead of preventing the reaction. Sodium metabisulphite and sodium formaldehyde sulfoxylate prevented the darkening, but accelerated the rate, of reaction. Buffering solutions to a more alkaline pH increased both the rate of darkening and decarboxylation and this effect was not prevented by antioxidants. Replacement of the air in ampoules by nitrogen reduced darkening but not decarboxylation.

It has also been reported that inert gases such as nitrogen, carbon dioxide and sulphur dioxide, which have been used as stabilising agents for menaphthone sodium bisulphite, actually accelerate its decomposition. Some preservatives are chemically incompatible with procaine penicillin and others affect its physical properties.

SUPPOSITORIES AND OINTMENTS

The type of base used for suppositories can significantly affect the absorption of medicaments. Similarly, ointment bases can affect both the stability and absorption of the active ingredients.

OPHTHALMIC PREPARATIONS

Reports of *Pseudomonas aeruginosa* infections from contaminated eye drops have been made in both Great Britain and the United States. Many preservatives have been tested for use in eye drops. The full paper contains references to many papers on the suitability of various preservatives for this purpose and extensive references to incompatibilities.

CONTAINERS

Glass containers can release alkalinity causing decomposition of many medicaments, especially alkalis. Many injections also have narrow ranges of pH for stability and must be packed in glass containers complying with a limit for alkalinity. It is well known that some types of glass can liberate lead to alkaline hydroxides, ammonium acetate solutions and similar substances. It has now been reported that cream of magnesia can dissolve arsenic from some glasses.

Schou has shown that non-resistant glass may have a layer of copper on its surface but this can be removed by routine cleaning. Silicon and boron can also be released from glass. If chromates are used for cleaning glass, chromium ions may be absorbed by the glass with subsequent release to solutions of medicaments.

The absorption of metal ions from glass containers may be responsible for the decomposition of many substances including adrenaline, vitamins and procaine hydrochloride. It has been shown that sodium amino-salicylate forms chelates with many metallic ions and this causes marked reduction of its antituberculous action.

The liberation of flakes of glass is a serious problem with containers for infusion fluids, especially those containing citrates and bicarbonates. Autoclaving the container with solutions of hydrochloric acid, citric acid or sodium metabi-sulphate will minimise this effect.

METAL CONTAINERS

Numerous examples of the deleterious effects of metals on medicaments have already been mentioned.

COPPER

The release of this metal from containers and apparatus and its effect on many types of preparations have been described in a valuable series of papers by Schou and his colleagues.

IRON

Iron causes discoloration of phenols and reacts with many substances. Even some types of stainless steel can react with methyl salicylate, oxymel of squill and other preparations containing acetic acid and with ammonia and senega mixture.

ALUMINIUM

Uncoated aluminium containers may cause blackening of tablets and in aerosol containers can cause decomposition of perfumes. Uncoated aluminium tubes are unsuitable for emulsion bases, ointments containing mercurial compounds or for substances with a pH outside of the range 6.5 to 8.0. Aluminum tubes coated with epoxy resins, however, are very unreactive.

TIN

Tin has been reported to be corroded by chlorides and by increase in acidity, but tubes of that metal have been used to contain morphine hydrochloride solution at pH 3.7 for several years without corrosion.

LEAD

Because of its toxicity lead should not be used for tubes for preparations for internal use. Lead tubes have also been reported to react with sodium alginate and with silver picrate. Tin lined lead tubes cause contamination of preparations containing electrolytes more readily than plain lead tubes.

PLASTICS

Most plastics are not completely impervious to water vapour and some loss may occur with concentra-

tion of aqueous products. Loss of volatile perfumes may also occur. Polyvinyl chloride transmits water vapour more readily than polythene.

Polythene tubes have the disadvantage of elastic recovery and hence cannot be rolled up like flexible metal tubes. As a result of this air is drawn into the tubes with increase in the chance of drying of the material and of oxidation.

Many plastics contain plasticisers and, since these may be extracted, their possible effect on the stability and toxicity of foods and drugs is very important. Data for the most commonly used plasticisers and other possible extractives from plastics have been given in a report from the British Plastics Federation. Many medicaments, such as phenols and esters, are incompatible with plastics.

CLOSURES

In addition to the well-known action of chemicals such as chlorine-liberating compounds and permanganates on corks, attention is drawn to the possibility of the contamination of cetrinide solutions occurring through storage in corked bottles.

Rubber closures are incompatible with paraldehyde and with nikethamide. They will absorb many antiseptics such as chlorocresol, phenol, phenylmercuric salts and thiomersal. They also absorb the antioxidant sodium metabisulphite. These effects can be minimised, but not entirely prevented, by preliminary soaking or refluxing the caps with the appropriate compound.

Rubber both absorbs water and transmits its vapour. It may also release compounds with potentially toxic effects and deleterious effects on the stability of medicaments. Zinc may be released to a sufficient extent to be physiologically objectionable or to cause insoluble particles by interaction with other constituents or with the container.

Plastic closures have also been shown to be superior to rubber ones from the points of view of physical properties and extraction of materials, but not from the viewpoint of absorption.

TESTING OF CONTAINERS

Several tests and standards have been suggested for containers. The ideal container is perhaps best summed up in the words of the U.S.P. requirements. "The container shall not interact physically or chemically with the drug which it holds so as to alter the strength, quality or purity of the drug beyond the official requirements."

Both the B.P. and U.S.P. include tests for limits of alkalinity of glass. The U.S.P. also contains tests for suitability of containers including chemical resistance to water and acids at 121°C. and for light transmission. The precipitation of alkaloids has also been used as a test of the suitability of glass for use in medicine bottles.

Storage tests at various temperatures are essential for full assessment of suitability of any container for any particular substance. After storage in the container under a variety of conditions, the product should be examined physically and chemically; the container should be examined for signs of corrosion. Mechanical strength can be tested simply by rough handling



- **EMULSIFIERS**

- **DETERGENTS**

- **ABSORPTION BASES**

- **FRAGRANCES**

- **SCIENTIFICALLY DEVELOPED
AND MANUFACTURED—
SPECIFICALLY FOR APPLICATION
IN COSMETIC FORMULATIONS**



or by a standardised drop test. Leakage can be tested by placing water in the container, storing at elevated temperatures and weighing at intervals. Permeability can be tested in the same way or by placing the container in an atmosphere of high humidity, varying the temperature and weighing at intervals.

TESTING OF CLOSURES

Numerous tests have also been suggested for closures, especially for rubber caps for injection bottles. These include tests for penetrability, fragmentation, self sealability, water extractive, alkalinity or acidity and compatibility with medicaments.

Penetrability is tested by the weight necessary to cause penetration of the cap by a needle under specified conditions and fragmentation by the number of particles found in solutions after the cap has been pierced a specified number of times.

Permeability can be measured by placing a strongly hygroscopic substance, such as anhydrous calcium chloride, in bottles sealed with the caps, storing them under conditions of high humidity and examining them for increase in weight. This method can be used as a general test of efficiency for containers and closures. It is made more stringent if the containers are subjected to numerous marked changes of temperature during storage.

Self sealability may be tested by forcing air into an inverted bottle and noting if any leakage occurs through the closure, or by placing methylene blue solution in containers sealed with the caps, immersing them in water and applying a vacuum. No signs of leakage should occur. For these tests a specified number of punctures is made in each cap.

For determination of extractive matter, caps are boiled or preferably autoclaved with distilled water and tests are carried out on the water after removing the caps.

Acidity or alkalinity is determined by measuring the pH of the solution. The weight of any residue from a given volume evaporated to dryness may be determined. Turbidity measurements, conductivity, determination of reducing substances and Nessler's test may also be carried out on the solution.

SUMMARY AND CONCLUSIONS

The numerous totally unexpected reactions outlined in this paper emphasise the importance of thoroughly testing all new formulae from the points of view of stability, efficacy and toxicity.

In addition, prolonged storage tests should be carried out under various conditions to guard against delayed reactions and to test the effects of containers and closures.

Since the free exchange of experiences in this field will improve the pharmaceutical services in all countries, pharmacists should be encouraged to publish their results. However small any individual example may seem, each has its importance in building up a complete knowledge of the properties of drugs and excipients. International exchange of ideas is especially valuable and symposia of this kind can be the means of disseminating knowledge over the widest possible field.

Use of radioisotopes in detergent and cosmetic research

M. F. NELSON

APPPLICATIONS OF radioisotopes to the problems of industry have been increasing at a tremendous rate during the past fifteen years. The Atomic Energy Commission estimates that industry is now saving nearly one billion dollars a year from the use of radioisotopes and predictions are that a saving of tens of billions of dollars within a decade will be realized (1).

Probably one of the most fertile fields for the use of radioisotopes is in the detergent and cosmetic industry. Use of beta and gamma gauging in production control has been undertaken, and use of radiation itself as a method of sterilization of products or product ingredients normally altered by bacteria is soon to be realized. Radiotracer techniques have found and are finding considerable usage as aids to product development and improvement. However, only a limited usage of radioisotopes has already been undertaken in comparison with the full potentiality of these materials as research or production tools, and corresponding industrial savings. The restricting factor in many instances has been a lack of knowledge of the uses of radioisotopes and also a lack of personnel trained in their usage.

Radiotracer techniques have already been used in a variety of ways in detergent and cosmetic research. Unfortunately the literature gives only a portion of the techniques which have been used to aid in the resolution of specific problems. This is due in part to the specificity of the particular study conducted or an unwillingness on the part of some industrial management to reveal techniques that may be direct aids to competitors in product improvement. Also some experiments which have been undertaken and techniques, which have been developed, are faulty or are not suitable for publication. Faulty procedures often

occur due to a lack of knowledge of radiotracer technique, as well as a tendency to formulate the method used to satisfy or substantiate advertising claims. Therefore, factors are often overlooked or ignored in tracer experiments that should be considered such as decrease in activity due to self or media adsorption of weak beta particles, failure to maintain constant geometry in a series of radioassays, apparent decrease in concentration of a tagged material due to penetration into an adsorbing substrate, decrease in radioactivity due to hygroscopicity of a tagged material or substrate to be evaluated, failure to correct for the decay of short half-life nuclides, metabolic decomposition of the tracer compound by animal bodies, and improper use of instrumentation. To illustrate with an actual experiment:

Several years ago a group of experimenters wished to evaluate the ability of various types of formulations in removing dirt from living skin. Amorphous carbon 14 mixed with soot to represent soil was rubbed into an area of animal skin and radioassays were taken. The "skin cleaners" were applied, wiped off the skin and another radioassay was taken. Initially it was found that results were very erratic and further investigation showed that in the assays, the Geiger tube was not being maintained a fixed distance from the skin—"The geometry of the system was not constant." At the "conclusion" of the experiment it was noted that one formulation showed high efficiency in removal of dirt as was shown by a marked decrease in radioactivity, although visually the skin area treated did not appear as "clean" as it did when treated with other cleansing agents. Subsequent investigation showed the formulation to cause a very marked decrease in radioactivity directly after it had been applied and even before the skin was wiped free of the cleanser and the dirt removed by the formulation. It was found that the formulation contained high concentrations of oils (in contrast to the

* Radiotracer Laboratory, Atlas Powder Company, Wilmington 99, Del.
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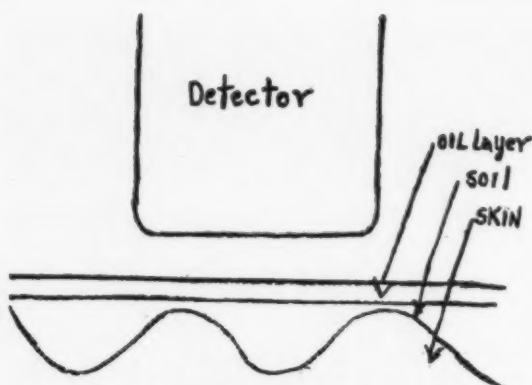


Figure 1

other formulations tested) which spread over the skin surface and were not readily removed by the wiping procedure. These oils acted as adsorbers for the weak beta particles emitted from the carbon 14 and the radioassay taken after wiping was low due to this adsorption. This effect of the oils is illustrated in Fig. 1. Consequently the formulation containing the oils appeared to be a better cleanser. Other formulas, not possessing materials which would act as adsorbing layers for the radioactive particles, gave a performance rated in proportion to their actual cleansing efficiency. There have been other instances in advertising literature where experimental techniques have cast doubt on the validity of the results.

It is obviously very necessary that good experimental design be used and the limitations of the studies be fully realized before attempts are made to draw broad conclusions on results obtained.

Cosmetic Research

With proper experimental design radiotracer techniques can be very valuable in furnishing solutions to existing problems. Use of carbon-14 and other



Figure 2

radioisotope tagged compounds has aided in determining the metabolic fate of additives to formulations in order to facilitate clearance of the materials by the Food and Drug Administration (2). This procedure of metabolic tracing is becoming common practice in the Pharmaceutical and Chemical Industries, and the F.D.A. generally desires the procedure as part of the evidence necessary before condoning the internal use of a compound by the general public.

Closely related to metabolic tracing are studies conducted on the lower animals to obtain information on products or product ingredients for substantiation of advertising claims or acquisition of knowledge.

To illustrate, the amount of retention of formulation additives such as tooth decay inhibitors as well as formulations themselves with respect to time and concentration of material used, has been followed (2, 3). An illustration is given in Fig. 2, showing the use of tracers to study the retention of this type of compound. In this case the carbon-14 tagged additive was incorporated a dentifrice formulation and by use of a trimmed mascara brush, the teeth of white rats were brushed with the formula in a manner similar to that used by humans. At specific intervals after treatment the animals were sacrificed, the oral tissues were combusted and subsequent radioassay showed the amount of retention of the test material.

Another example of this type of experiment is the tracer study conducted for a manufacturer of a vaporizing ointment used as a cold medication (4). Using tritium (radioactive hydrogen) tagged camphor and menthol in the ointment, experiments were conducted on dogs to determine the rate of inhalation of the vaporizing ointment.

Also studied was the duration of volatilization of the aromatic oils from the ointment base.

In studies conducted with carbon-14 tagged detergents on human hair, casein and gelatin, the effect of pH, solution concentration and exposure time as well as condition of the substrate (as altered by chemicals) has been determined (5). By exposing hair fibers to solutions of tagged detergents with subsequent preparation of autoradiographs, the mode of penetration of these compounds into the hair fiber has also been followed (5). It is pertinent here to point out that extreme care must be taken in the preparation of autoradiographs associated with human hair or skin. It is possible to cause "exposure" of the film by the hair fiber itself with no radioactivity present, because of chemical reaction between the sulfur containing proteins present in the hair (such as cystine and cysteine) and the silver salts in the photographic emulsion.

The adsorption of sulfur 35 tagged dyes and the penetration of hair fibers by sodium bromide (tagged with bromine-82) has also been thoroughly investigated (6). Studies of this type are of interest in cosmetic chemistry since much can be learned of the additives used in preparation of hair dyes and rinses.

In respect to skin studies, the literature contains many articles which describe the use of radioisotopes as tools for the evaluation of materials on both human and animal skin. In most instances, radioisotopes

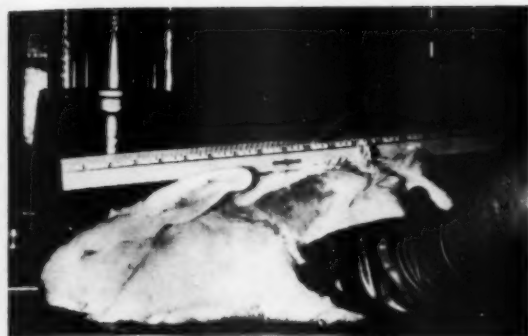


Figure 3



Figure 4

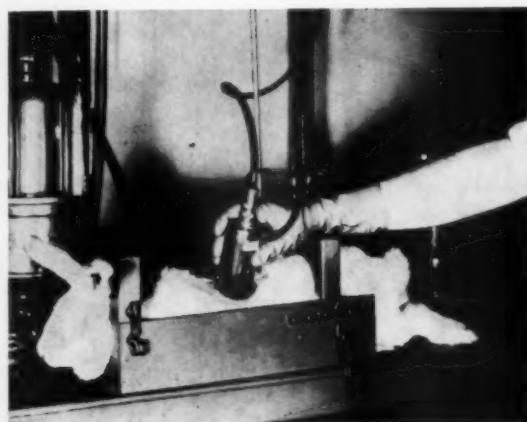


Figure 5

were used because there was no alternate method of obtaining the answers desired. Since attempts to extrapolate *in vitro* or test tube results in terms of what would occur in the presence of living cells often results in failure, studies were undertaken directly on living tissue.

Numerous studies have been undertaken on the skin adsorption of carbon-14 tagged bacteriostats. Studies have been performed in respect to the adsorption of hexachlorophene from soaps onto the skins of rats, rabbits and humans (7, 8, 9, 10). The effect of repeated washing of living skin with soaps containing the tagged bacteriostat was investigated, and build-up of the bacteriostat was followed. Also studied was the effect of concentration of bacteriostat on build-up as well as the rate of removal of the



Figure 6

compound from skin tissue by washing with soap containing no bacteriostat.

Figures 3, 4 and 5 pictorially show how this type of study was conducted on animal skin. These pictures, taken during an actual experiment, show stepwise the procedure used. Figure 3 shows the shaved skin of the rabbit. The section on the upper back was generally used as the test site since a reasonably flat area could be found on which to position the special radioassay device. The actual assay site on the rabbit skin was marked with India Ink. Figure 4 illustrates the washing of the skin with the soap containing the tagged bacteriostat using a small section of a cellulose sponge (used since it adsorbed little or no bacteriostat). After the skin was rinsed with water and dried with a hair dryer, radioassay was undertaken as shown in Fig. 5 using the special "Flowgas Cell" devised for this purpose. Figure 6 shows a closeup of this cell which has been described previously in the literature (7). The cell is advantageous over a thin and window Geiger-Mueller tube in that it possesses much greater sensitivity. Furthermore, since it is placed in intimate contact with an adsorbing substrate such as skin, the distance from detector to radioactive source is fixed and geometrical corrections are not necessary.

In all cases normal chemical analysis of the bacteriostat, even *in vitro*, was difficult, sensitivity was poor and many other compounds interfered with the analytical procedure. However, with radiotracer techniques, adsorption values were easily obtained.

The adsorption and retention of carbon-14 tagged emollients from lotion formulations by human and animal skin has also been studied (5, 11, 12). In some cases special radioassay devices were designed in order that proper experimental procedure was used and that results obtained were valid. Cold cream labeled with carbon-14 has also been used in studies and has been found to penetrate only very slightly into the skin (12).

Evaluation of astringents used as antiperspirants in lotions and sprays has also been undertaken by

means of radiotracer methods. In one particular technique developed (13, 14), the astringent properties of antiperspirants were studied by first treating live frogs with the astringent. The animals were pithed and the permeability of the frog skin was measured by determining the rate and amount of iodine-131 which migrated through the membrane.

Figure 7 illustrates the experimental method used for determining the amount of radio-iodine migration. Radioassay has been undertaken by measurement of the beta particle emission from iodine-131, or by determining the gamma activity of a portion of the water in the tube using a gamma scintillation spectrometer. The rate of iodine migration through the skin is subsequently correlated with the effectiveness of the astringent. There is no other satisfactory method at the present time available to industry that will effectively evaluate astringents in respect to antiperspirant properties.

With slight modifications this technique has been used to study other classes of compounds. To exemplify: by use of tritium in the form of tritiated water in a manner very similar to the preceding procedure, the effect of such materials as "barrier creams" may be evaluated in respect to their properties of preventing penetration of water through a skin membrane. Again these are examples where radiotracer techniques have been devised to furnish results that are either very difficult to obtain or are unattainable by other means.

Many studies are reported in the literature for determining retentiveness in the oral cavity and on human teeth of various agents from dentifrice formulas and from solution. Among the radiotracers used have been calcium-45, iodine-131, zinc-65, phosphorus-32, chlorine-38, fluorine-18, sulfur-35, rubidium-86, potassium-42, sodium 24, and carbon-14 labeled compounds such as urea, nicotinamide, thiourea and acetamide. The length of retention, rate and depth of penetration into tooth structure as well as localization in defects in tooth surface have been studied.

The increased use of fluorides in dentistry has prompted many investigations with radiotracers to determine conditions necessary for optimum adsorption and retention of this material on teeth. With the

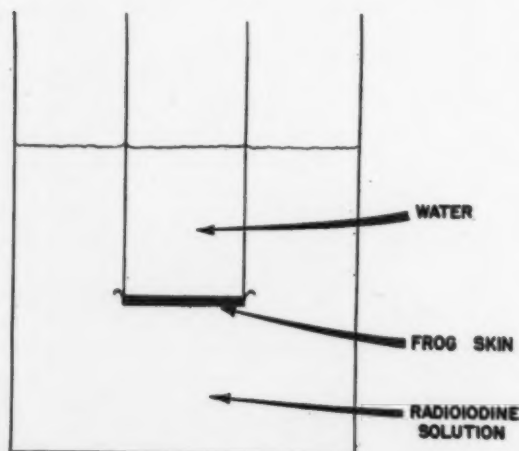


Figure 7



Figure 8

advent of a procedure for reactor production (15) of fluorine-18, many studies have been conducted with this tracer. The effect of tooth structure as well as the type of fluoride used have been investigated in several formulations (16). Dental fluoride adsorption has also been studied by tagging the cation (7), and in this manner the relation of cation to anion adsorption could be determined. Autoradiographs also have been prepared showing localization of the fluorine-18 in defects in tooth surface (16).

Figure 8 illustrates use of an autoradiograph to determine the localization of fluorine on an intact human tooth. The tooth in the right portion of the figure, possessing natural surface defects such as cracks, lesions and spots when treated with radio fluorine, gave the autoradiograph shown on the left. By inter-comparison the relative uptake of fluorine in specific sites is clearly demonstrated. Resolution is only fair due to the fact that fluorine-18 is a positron emitter and the tooth was covered with aluminum foil to cause positron annihilation before these particles came into contact with the film. It has been found by experience that if annihilation of the positron occurs in the film emulsion, very poor resolution is encountered with autoradiographs.

Although analytical methods for the analysis of fluorine were available, the analysis was only moderately sensitive and again interference by other ions occurred.

Detergent Research

In respect to soaps and detergents as a class of compounds, primary use of radioisotopes has been in reference to "detergent evaluation" or determination of the detergent's ability to remove soil from specific substrates. In usual studies employed to evaluate detergents the procedures used are somewhat lengthy, require bulky equipment and results obtained are dependent on a fairly close control of experimental techniques. In some instances with certain substrates, evaluation is very difficult. Therefore a considerable number of articles have been published in an attempt to find more suitable methods for detergent evaluation. Methods using radioisotopes have been reported and appear to be excellent.

Studies have been undertaken by several investigators to determine the efficiency of detergents in removing oil soils from metal surfaces (17, 18, 19, 20, 21). In these studies, carbon-14 tagged compounds such as fatty acids and their derivatives have been used. By use of carbon-14 tagged stearic acid as the

oily soil, the properties of several metal cleaning solutions have been investigated (18, 19). The effect of sequestrants on detergent action has also been studied in respect to the removal of dirt containing fission products as tagging agents, especially for evaluation of detergency in respect to radiological decontamination (21). Other experiments have involved the synthesis of radioactive bacteria by feeding the bacterial cultures radioactive phosphorus 32, in order to study the ability of various detergents to remove bacteria from dishes and utensils associated with the preparation and serving of food (22, 23).

The studies of primary interest to manufacturers of soaps and detergents have been those which have developed techniques whereby "detergent evaluation" may be undertaken routinely in respect to removal of soil from textile fibers. In this respect extensive studies have been undertaken into the preparation of radioactive soil that would truly be representative of the usual soil found on cloth (24, 25). Among the radioactive materials used as tagging agents for soil have been fission products, carbon-14 and calcium-45.

One complete technique in use by several companies for detergent evaluation utilizes an oily paste prepared from radioactive carbon-14 lampblack mixed with mineral oil as the soiling agent (26). In the test, cloth is soiled by first picking up a small amount of the oily soil on an etched plate and then mechanically rubbing traces of the soil onto Indianhead muslin swatches. The swatches are radioassayed to obtain the amount of radioactivity representative of the amount of initial soiling present, and are then washed in a miniaturized "washing apparatus" with the detergent to be tested. The swatches are dried and again radioassayed and the amount of soil remaining is thus determined. Soil redeposition in the presence of the test detergent may be studied by including an unsoiled swatch in the washing apparatus and determining the amount of radioactivity picked up by this swatch. The commercial availability (27) of cloth swatches soiled with carbon-14 tagged lampblack, protein or fat has further simplified the technique.

This test method is simpler, reasonably inexpensive, more sensitive and faster than usual evaluation studies utilizing reflectometers or visual inspection as an index of cleaning ability. Furthermore, changes in the surface of the cloth which can cause changes in reflectance have no effect in the radiotracer technique. An additional advantage is that because of the small quantities of all substances used, very small amounts of detergent may be evaluated. Further improvement of the technique has recently been made with the preparation of a radioactive clay as a soiling agent (28). This clay is prepared by the addition of calcium-45 or strontium-90 salts to normal clay with subsequent isotope exchange and the formation of "tagged" clay.

Determination of quantitative adsorption soaps and detergents onto textile fibers has also been undertaken. The adsorption of sulfur-35 tagged sodium lauryl sulfate and sodium alkyl benzene sulfonates and carbon-14 tagged sodium palmitate has been studied. These studies were undertaken not only to correlate detergent action and soil removal properties with

adsorption, but also to study the effect of detergent adsorption on soil redeposition.

Other uses are known for radiotracers in the detergent and cosmetic field. The complete rheology of systems involving the addition of one component to another have been studied. By use of iodine-131 or sodium-22, the addition of "builders" and additives to detergent bases has been followed in respect to motor speed, position and angle of stirring, amount of additive, temperature, time of stirring and shape of vessel. With use of a scintillation counter and gamma emitting isotopes (so that absorption corrections are not necessary), techniques have been developed that are faster, more accurate and far more sensitive than usual analytical methods.

Stability of emulsions can also be evaluated using radioisotopes (19). By tagging one component in an emulsion and by radioassay along the vertical side of the vessel containing the mixture, the gradient of radioactivity obtained from the surface of the mixture to the bottom is an excellent index of homogeneity. Quite often, long before visual signs of separation occur, the gradation of radioactivity has shown the emulsion mixture to be separating. Therefore the technique permits quicker evaluation of lotions and creams in respect to their stability toward separation.

Summary

Uses of radioisotopes in detergent and cosmetic research is demonstrated, with specific examples being given of radiotracer techniques applied to various problems.

In the examples shown, the use of radioisotopes was stimulated by either lack of routine analytical techniques suitable for assay of the desired material or very high costs of experimental methods by use of these techniques. Manpower hours were saved and valuable research knowledge was gained which aided in product development and improvement.

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Instrumental analysis of essential oils and related products.

No review of the present status of instrumental analysis in the United States would be complete without a tribute to those outstanding chemists in the field of essential oils who labored without the benefit of our modern instruments. A list of these early workers must include the great names in terpene chemistry: Wallach, Tieman, Semler, Tilden, Simonsen, Kleber and Powers.

It is true that these chemists made good use of those instruments which were then available, notably the polarimeter and the refractometer. Thus, in the older literature, it was not uncommon to see calculations of molecular refractions to prove (or disprove) the presence of a double bond in a carefully purified isolate.

Let us never underestimate the herculean task faced by a chemist in those days in his attempt to establish the molecular configuration of a terpene or terpene derivative, and the almost impossible feat of unraveling the secrets of the sesquiterpene series.

Such work required the development of unique and elaborate chemical techniques. To separate geraniol and citronellol, Tieman developed a method based on the relative resistance of Citronellol to dehydration with Phosphorus Trichloride. To separate isomeric ionones, it was necessary to resort to the solubility differences of certain derivatives. The introduction of the hydrochloride derivatives for the terpenes by Tilden represented a great step forward. Ozonolysis, followed by identification of the fragments, was commonly used to show the position of double bonds.

Nevertheless, many questions could not be answered with certainty. Were the terminal endings of terpene alcohols represented by isopropenyl, or by isopropylidene groupings? Did the drastic chemical treatment shift the double bond?

The chemist of today owes a debt of gratitude to the instrument manufacturers. Theirs has been a constant effort to improve, to simplify where possible, to produce a practical instrument for our use. And they have been eminently successful.

The commercial production of today's instruments has become a highly competitive industry. Competition has been responsible for instruments of high

quality, versatility and good reproducibility. Manufacturers continue to emphasize the advantages and applications of their products to laboratories throughout the country, thus making all chemists aware of the importance of instrumentation. It is not unusual for an instrument manufacturer, prior to the development of a new model, to survey laboratories known to use similar instruments in order to learn of the shortcomings of existing equipment. Thus, improvements and new ideas are incorporated in instruments to satisfy the general needs and requirements of industry. In this way, the instrument users themselves contribute to the improvement and development of the modern instrumentation. The recent production of a moderately priced double-beam IR instrument for bench work has aided in the expansion of IR in many laboratories. The advent of "assembly line" models of gas chromatographs, which are of relatively low cost, has introduced instrumentation to many of the smaller laboratories.

The essential oil industry has yet to utilize fully instrumental analysis. Several major companies in this field, however, have set up instrumental laboratories and are using to advantage infrared, near infrared,

This paper was read at the Indian Standards Convention, December 31, 1959, during the session on Latest Techniques in Chemical Analysis. The authors are Edward E. Langenau, vice president and director of analytical laboratories, Fritzsche Brothers, Inc., and James A. Rogers, Jr., director of instrumental laboratories for the same firm.



Edward E. Langenau



James A. Rogers, Jr.

American Perfumer & Aromatics



SPEAKING FROM ENGLAND

JOHN BERNARD WILKINSON, B.A., B.Sc., F.R.I.C. became Research Manager of the Toilet Preparations Development Unit of Unilever Ltd., in June, 1957. Before taking up his present appointment, he had been, successively, technical director of Joseph Watson and Sons Ltd., Leeds, and technical director of A. & F. Pears Ltd., Isleworth. He is a member of the Council of the Society of Cosmetic Chemists of Great Britain and was one of the two British delegates to the International Federation of Societies of Cosmetic Chemists.

Glycerine

London

IN SKIN CREAMS AND LOTIONS

In considering the formula for a toilet product, we must look at the product as a whole, including the package, against the envisaged uses of the product as a whole, not just at a single ingredient in isolation. To do otherwise can lead to trouble, both in the plant and in the home.

This becomes very obvious when consideration is given to a 'special ingredient', such as a germicide, added to a product of basically simple use pattern such as a soap, toothpaste or a shampoo. At one extreme, the main object of the operation is to provide a vehicle for the germicide or other special ingredient; in this case the product is built round the active principle and is designed to permit its application and working under optimum conditions. At the other end of the scale, the formula is modified as little as possible from the optimum for its main function so as to permit it to carry the 'plus' ingredient to the greatest advantage. Between these extremes lie clearly many compromises.

Both extremes point the moral that, although one may begin by considering the virtue and properties of one ingredient, the good formulator soon starts thinking in terms of the product as a whole in relation to its projected use. Therefore when we come to the question of glycerine in skin creams it is first necessary to consider the products in question and their uses. For the purpose of discussion, I have confined myself to the product types in Table I, taken generally from European practice, and covering in principle most products generally considered as skin creams or lotions.

Examination of this table, together with the 'characteristics' in Table 2, coupled with the general principles of

cosmetic formulation, shows several common ideas behind the formulation of all these creams and lotions. Now consider the role of glycerine as a part of the formula for these product types in the light of the requirements for consumer use. Only when this has been done, is it possible to embark on the preparation of type formulae for the different product classes in a logical manner.

Product Properties

1. Drying-out. There is a general requirement for slow drying-out of the product in the jar. For the general purpose creams, the small sizes of which are often packed in slip-lid cans, this is made possible by use of a water-in-oil emulsion. Typical results below obtained in our laboratories show both the effect of glycerine in a water-in-oil cream and the vast difference in behaviour of two different oil-in-water creams.

Cream Type		Weight loss % in 1 week at 37°C.
General purpose cream (w/o) A	(no glycerine)	6.2
" " " "	A (+4% glycerine)	3.9
Compare with:		
Vanishing cream (o/w) B		34.8
" " " " C		74.0

But, for all types of cream, storage before and during use demands minimisation of water loss. However perfect the closure design, we have to contend with casual deficiencies and incorrect reclosure by the user. Under the worst conditions, cavities can be produced in some types of cream, no-

TABLE I—THE PRINCIPAL TYPES OF SKIN CREAM AND LOTION

Product	Emulsion Type	Phase Proportions		Main Uses
		% oil phase	% water phase	
Foundation Cream	oil-in-water	15-30	70-85	As a foundation for face powders. May also provide colour by means of pigments or soluble dyes.
General Purpose Cream	water-in-oil	35-45	55-65	Foundation, cleansing and skin care; a compromise in efficiency between its various uses.
"Nourishing" Cream	water-in-oil	35-45	55-65	Includes creams containing special ingredients such as hormones.
Cleansing Cream	water-in-oil or oil-in-water	50-65	35-50	Removal of make-up and cleansing of face at night. Normally used in association with tissues.
Night Cream	water-in-oil	65-90	10-35	Overnight treatment, particularly as a corrective for dry skins. May also be used for massage.
Cleansing lotion/milk	oil-in-water	10-30	70-90	Removal of make-up during day. Applied on cotton wool.
Hand cream/lotion	oil-in-water	5-20	80-95	Treatment of dry hands or those damaged by work.
Skin lotion	—	—	100	Astringent or emollient; also a source of fragrance.

tably the traditional, aerated, vanishing cream and in some others a surface crust may form, which then restricts further water loss but is objectionable and unsightly.

A humectant can play a part in reducing these troubles, but do not exaggerate its possibilities. Typical water phases contain nowhere near the glycerine content required for them to be in equilibrium even with higher atmospheric humidities. Added glycerine can however restrict transfer of water vapour to the near-saturated atmosphere above a partially-closed jar and undoubtedly reduces the rate of loss of moisture. Henney, Evanson and Sperandio¹ have published comparative figures for several humectants and point out the influence of water content, humectant content and humidity. Evidence has been given by Rovesti and Ricciardi² that sorbitol is more effective in reducing rate of loss of water, but it must be remembered that this is only one of the properties required from a humectant.

In all creams some degree of control in use is necessary, despite the fact that the water content is ultimately required to disappear. In use, the water is either given up to the skin to restore lost moisture (see below), or, having done its job of giving fluidity and spreadability, it dries out onto the atmosphere; in neither case is it desirable for the water phase to disappear too quickly.

2. Skin humidification. The pleasure of a smooth, supple skin and the dislike of a dry-feeling hard skin have been appreciated by the non-experts from time immemorial. The credit goes to Blank³ for stating clearly the connection between the feel of the skin and its water content. For finding simple practical ways of alleviating the less serious conditions, credit goes to the cosmetic industry, but, it must be admitted, largely by empirical methods. But to-day, with new knowledge, heavy emphasis is being placed by the beauty profession on 'moisturising' with a partial understanding of the old empiricism.

The presence of a humectant in the water-phase would be expected to stabilise the water content during the later stages of absorption or drying-out; and glycerine should fulfil this role well, although satisfying experimental proof is still lacking. The difficulties of work in this field have been well brought out by Powers & Fox⁴ and we must surely support their conclusions that 'a great deal of additional study and work will be required'.

3. Lubrication. With all creams and lotions, good spreading is required not only initially but also sometimes during a prolonged working-in. Aqueous glycerine of all strengths provides a subjectively very acceptable medium and has the added advantage of good compatibility with other ingredients; in particular it can help to prevent 'balling' and 'rolling' when these properties are not desired⁵ as suggested by Kalish. Furthermore, in lotions, glycerine can provide unctuousness

without oiliness; this is of particular advantage in some more specialised products such as after-shave lotions and sunscreens.

4. Viscosity of Water Phase. The lubricant properties of glycerine solutions are allied to those of providing suitable increases of viscosity. For aqueous or aqueous-alcoholic lotions, the viscosity is often required to be controlled at a high enough level for ease in use. In emulsions, the viscosity of the continuous phase plays a part in providing a satisfying 'body' to the product, and especially in the thinner lotions and milks, glycerine can provide this adequately.⁶

5. Emulsion Stability. In emulsified products ingredients giving lubricant properties or increasing the viscosity of the water-phase must not prejudice emulsion stability. Glycerine certainly does not do so. Indeed we believe that in some complex emulsion systems, it may positively promote stability, especially where monoglyceride is used as one of the emulsifiers. In less complex emulsions, increase in stability through glycerine addition may be due solely to an increase of viscosity at the interface.

6. Perfume Stability. All ingredients in a cosmetic must be selected to have the minimum deleterious effect on perfume stability. Glycerine is certainly sufficiently non-reactive to meet this neutral requirement; indeed, probably by virtue of its solvent powers and low vapour pressure, it has some fixative action and is a valuable adjunct for this reason alone.

7. Safety and Mildness. Of necessity no ingredient can be employed which is not safe on the skin in the proportions used, both as in the formula and after drying-out or absorption (a point which is often overlooked). Glycerine meets this requirement.⁷

The hygroscopic nature of pure glycerine has been held to be a disadvantage on the grounds that it might draw water from the upper layers of the skin in order to reach its appropriate equilibrium. (See Powers & Fox⁴ and Rovesti & Ricciardi²) This seems to me irrelevant since the equilibrium is always approached from the other side.

The relative humidities of the atmospheres in equilibrium with various strengths of glycerine/water solutions are given in a graph by Miner & Dalton,⁸ from which the following round figures are taken:-

% glycerine	Relative Humidity
65	70%
80	50%
90	30%

Thus for instance, even in a 70% R.H. atmosphere in the immediate neighbourhood of the skin, the glycerine solution would need to be initially stronger than 65% before it became hygroscopic. Such concentrations are never used in practice,

TABLE 2—FORMULATION OF SKIN CREAMS AND LOTIONS

Product	Required Characteristics	Typical Ingredients		
		Oil Phase (%)	Water Phase (%)	Emulsifier
Foundation Cream	Must spread easily and give a smooth, non-oily film to hold powder; may be tinted or untinted.	(1) Traditional "vanishing cream" type Stearic acid 13-20 (2) Neutral Formulation Waxes or cetyl alcohol 5-10 Mineral oil 10-15	Glycerine 8-10 Glycerine 2-5	Potassium or triethanolamine stearate formed in situ. Polyethylene glycol/cetyl alcohol ether.
General Purpose Cream	Must spread easily and "moisturise". Has fairly fluid oil phase but must not be subjectively too "greasy". Must not dry out too easily if to be packed in slip lid can.	Mineral oil and petroleum jelly 25-30 Lanolin 2-3	Glycerine 5	Wool wax alcohols and/or cholesterol. Nonionic emulsifiers
Nourishing Cream	"Moisturising" quality required, especially if special ingredient is in water phase. Formula must be compatible with any special ingredients used.	A wide range of formula is possible, but generally avoiding extremes. Many of these products may have bases similar to the general purpose creams.		
Cleansing Cream	Must spread easily, be an efficient cleanser and remain oily on skin. Must not dry out in jar as large packs are often used.	Mineral oil 25-35 Lanolin 5-10 Waxes 3-10	Glycerine 5	Wool wax alcohols Nonionic emulsifiers

and until we have further knowledge of dynamics of water transfer process at the skin surface, there seems no reason for doubting the traditional safety of glycerine.

8. Antiseptic Properties & Preservation. Many toilet preparations to-day have antiseptic or even germicidal ingredients added to them—a matter on which comment must be left for another place and time—but the addition of preservatives is almost universal; the need for this has largely arisen from improvements in cosmetic formulation. Many of the older creams made with saponaceous emulsifiers needed relatively little preservation because they provided a mildly hostile environment for most microorganisms; unfortunately these creams were also mildly irritant to a few sensitive people. Products to-day tend to be "kind to skin—kind to bacteria", and we have thus new and sometimes severe problems of preservation on which much has already been written elsewhere. (Particularly see 'American Perfumer' Documentary on Preservatives and Anti-oxidants, January & February issues, 1959.¹²)

It is thus a desirable feature of any ingredient chosen for other purposes that it shall also have some antiseptic or bacteriostatic properties.¹³ Barr & Tice⁹ have shown that most humectants have such properties but only at concentrations high enough for a presumed cosmetic action to be effective, such concentrations occur in toothpaste formulae, but not usually in skin cream water phases. However we do know that glycerine does not inactivate preservatives, which is more than can be said for many ingredients.¹³

Product Formulation. The principles of product formulation require us to select ingredients firstly for their positive qualities given the required properties and secondly for their neutral qualities of non-interference with the remainder of the

system. It thus becomes clear that it is advantageous to include glycerine in substantially all skin creams and lotions at levels to be chosen according to requirements. Omission of glycerine or use of other humectants and/or thickeners can provide different properties which may in some particular instances be desirable, even at the expense of other desirable qualities, but these specialised cases cannot be dealt with in a generalised study such as this.

Every cosmetic chemist will have his own formulae for the products with which he is particularly concerned, but it may be helpful to give skeleton formulae for each of the product types given in Table I if only to indicate the levels of glycerine recommended. Most of these are to be found in various forms in published sources and all are well established in practice with their appropriate perfumes and colours; all formulae must always be read with the reservation that perfumes and colours, as well as details of manufacturing technique, can affect some of the properties, notably stability, appearance and texture. Many variations are possible on these skeletons particularly by use of various branded emulsifiers and emulsifier combinations, for information on which current literature should be consulted. The figures given in Table 2 are only quoted to indicate the different glycerine levels which have been found to be effective in practice and not to provide a complete cosmetic formulary.

SUMMARY. Consideration has been given to the user properties and requirements of the principal categories of skin creams and lotions. It has been shown that the properties of glycerine as an ingredient are such as to make its inclusion very desirable in products of this type. An indication is given of suitable proportions of glycerine in various types of product. A list of references is available upon request from the Glycerine Producers' Association.

TABLE 3—TYPICAL INGREDIENTS

Product	Characteristics	Oil Phase	Water Phase	Emulsifier
Night Cream	Firm consistency required if used as massage cream; should have "moisturising" properties.	Petroleum jelly 20-30 and mineral oil Waxes 10-15 Lanolin 5-10	Glycerine 2-5	Beeswax-Borax
Cleansing lotion/milk	Viscosity depends on intended method of use. Must remove make-up efficiently and should not leave the face too greasy.	Mineral oil 5-25 Waxes 1-5	Glycerine 3	T.E.A., stearate or oleate in situ.
Hand cream/lotion	To be "moisturising" and must spread well and not leave hands too greasy.	Lanolin 1-2 Cetyl Alcohol or waxes 2-5 Mineral oil 3-10	Glycerine 5-8	T.E.A. stearate
Skin lotion	Good control of spreading may be required in addition to specific properties such as emollience, astringency, etc.	—	Glycerine 5-25 Alum if required Gums to required viscosity	—

In all cases, water to 100 after inclusion of emulsifier, perfume and preservatives.



IN TOILET GOODS

EACH YEAR 30,000,000 POUNDS OF GLYCERINE...

... are used for toilet goods and pharmaceuticals. Glycerine and its derivatives are found in virtually every type of personal care product. They are widely specified ingredients in the U.S.P. and N.F. All, for good reason: Glycerine's versatile combination of properties is unmatched in any other chemical.

IMPARTS BODY. Glycerine acts as a lubricant and can provide unctuousness without oiliness. It is an exceptional bodying agent for liquid preparations, syrups, emulsions and gels.

COMPATIBLE. The solvent power, miscibility and compatibility of glycerine give the formulator an extended range of materials with which to work.

MOISTURIZES. Glycerine attracts moisture and holds it in the product and on the skin. It promotes softness, flexibility, creaminess and long shelf life in countless formulations.

NONTOXIC. The safety of glycerine on and in the human body has been established through generations of use as well as by supporting clinical data. Glycerine and mono- and di-glyceride emulsifiers have been listed by the F.D.A. as "generally recognized as safe" as used in foods.

GOOD REASON FOR GLYCERINE

IN DRUGS



SWEET. Glycerine imparts a desirable degree of sweetness without clashing with other ingredients. Perfumes or flavors remain "true to type."

COMPLETE INFORMATION

Our 18-page booklet, "Glycerine Properties • Reactions • Performance", offers full information to anyone interested in glycerine. We'd be glad to send you a copy. Address: Glycerine Producers' Association, 295 Madison Avenue, New York 17, N.Y.



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ultra violet and visible spectrophotometry, vapor phase chromatography and, in a few cases, nuclear magnetic resonance and even mass spectrometry. One of the reasons for the hesitancy to accept instrumentation in the essential oil industry has been the fact that the human nose had a greater minimal sensitivity for odor compounds than the analytical instrument. However, present day VPC apparatus, utilizing the new ionization detectors and columns, boasts of extreme sensitivity—sensitivity which can match or even better organoleptic concentration requirements. Thus, for the first time, we can see, on a chromatogram, the materials responsible for overall odor. However evident they may be on a curve, it is still a problem to identify the peaks of those items which are vital contributors to the odor.

We feel that mention should be made of the facilities in the United States that are offered to instrumentalists in the essential oil field, enabling them to keep abreast of recent advances and techniques.

The *Society for Applied Spectroscopy* recently has incorporated all of the spectroscopy societies throughout the country into one group with various sections. This Society unites all instrumentalists working with absorption, emission, VPC, etc.; it is not restricted to any particular area of chemistry. Its bi-monthly publication, *Applied Spectroscopy*, is an excellent source of valuable information on instruments and techniques. It conducts annual symposium and instrumental exhibition in New York.

The *Coblentz Society* has been founded for those interested only in infra red. It is attempting to collect and distribute spectra of pure compounds submitted and screened by its members. It is also responsible for the infra red quantitative data appearing in *Analytical Chemistry*.

The *Pittsburgh Conference*, a joint effort of the Analytical Chemistry Group, Pittsburgh Section, American Chemical Society and the Spectroscopy Society of Pittsburgh, is a week-long meeting with the finest nationwide exhibition of analytical instruments and equipment, both foreign and domestic. An excellent program of papers is presented before the various sections which include:

Analytical Chemistry—General, Inorganic, Nucleonics, Fundamental Aspects, Electro-analysis, Analysis of Complex Mixtures, Quantitative X-Ray.

Infra Red—General, Near Infra Red Symposium, Group Frequencies Symposium, Recent Advances and Trends (For Infra Red 15-40 microns), Instrumentation.

Gas Chromatography.

Mass Spectrometry.

The *Essential Oil Association of U.S.A.* is well known for its standards and specifications for essential oils and aromatic chemicals. The Scientific Committee has established a Subcommittee for Instrumental Methods in an attempt to establish analytical specifications by instrumentation for essential oils and related products. This committee has worked very diligently and has pooled the knowledge and technique of the various laboratories of the members to:

Determine reproducibility and study standardization of instruments;

Publish general quantitative methods for components of essential oils and aromatic chemicals;

Describe specifications for individual components of these products.

These publications will be made freely available not only to the E.O.A. members, but also to other interested parties. Such studies include free hydroxyl in esters by Vapor Phase Chromatography, Near Infra Red, Infrared Red; free hydroxy-citronellal in the Dimethyl Acetal by Infra Red and VPC; Linalool contents of Bois de Rose and other oils by VPC, Infra Red and Near Infra Red; separation of Ionone and Methyl Ionone isomers by VPC. In all cases, recommendations will be made for adoption of the best of the methods studied. To our knowledge, this is the first attempt to do this type of work and the first attempt to pool instrumental information within one industry.

Other services that are available to essential oil instrumentalists are the standard IR and UV spectra of pure materials distributed by governmental and private groups. These groups include:

American Petroleum Institute
Project 44 (2,000 spectra);
Sadtler Research Laboratories Catalog
(12,000 spectra);
National Research Council,
National Bureau of Standards
(1,000 spectra).

Because of the large number of spectra now available, punch card methods are employed for identification and sorting. The American Society of Testing Materials has coded on IBM cards the spectral data contained in the three catalogs mentioned above and also that contained in the DMS System (Butterworth Publications, London). The ASTM-IBM decks total about 14,000 IR and 8,000 UV cards.

Leading American universities and colleges, such as:

Massachusetts Institute of Technology
Brooklyn Polytechnic Institute
Canisius College
Boston College
Rensselaer Polytechnic Institute

Offer intensive courses of training in the use of various instruments.

Let us now consider some of the individual instruments which have proven to be such an aid to the chemist in the field of essential oils and related products.

1. SPECTROPHOTOMETRY

Spectral instrumentation in our field has been applied extensively to quality control, research, development and production. For convenience, we shall consider some of these applications on the basis of the range of radiation.

A. ULTRA VIOLET

The UV range (200 to 380 m μ) is very sensitive to unsaturation; e.g., aromatic, olefinic, carbonyl components. As such, it can be used extensively in the study of essential oils; it has been observed that

three out of four essential oils show UV absorption. The problem, however, is to determine the component or components contributing to the UV peaks.

Following the work of Sale (J.A.O.A.C. 36, 112; 1953) on Lemon Oils, many laboratories have applied his technique to other citrus oils. It is apparent that the natural coumarins are mainly responsible for the UV peaks reported. There are some fifteen substituted coumarins present in citrus oils; all absorb in the UV range. Compounds such as citral (and other conjugated aldehydes), p-cymene, phellandrene, dimethyl anthranilate and conjugated dienes cause the citrus oil curves to go off scale below 270 m μ , but we can read the less intense bands around 320 m μ which are due mainly to the substituted coumarins. Stanley and Vannier (J.A.C.S. 79, 3488; 1957) isolated seven coumarins in coldpressed lemon oil, all of which proportionately contribute to the UV peak at 315. Differing proportions of coumarins are responsible for the shift of the peak maxima in other citrus oils from 315 m μ for lemon oils to 319 for grapefruit, 320 for bitter orange, 325 for tangerine and 329 for sweet orange. Dimethyl anthranilate in mandarin oils is partially responsible for the formation of a doublet in some oils and shifts the peak to within the range of 323 to 343 m μ depending on the concentration in the individual oil.

Piperine, the important pungent constituent of black pepper, can be quantitatively determined in the oleoresin by UV. In the past, this alkaloid has been determined by the Kjeldahl method; other nitrogenous components may interfere, however, in the older method.

Capsaicin, the pungent principle of red pepper, can also be determined with the aid of UV.

Aldol condensations (such as the Claisen-Schmidt) can be readily followed by UV absorption. This is important not only for evaluation of a product, but also for control during manufacture. For example, the concentrations of unreacted citral (measured at 237 m μ) and of the formed pseudoionone (measured at 292 m μ) are easily determined. After cyclizing the pseudoionone, alpha ionone will absorb at 228 m μ and beta ionone at 296 m μ ; however, any unconverted pseudoionone will interfere.

For the essential oil chemist, UV methods are valuable in many quantitative determinations:

safrol in saffron oil;

methyl salicylate in wintergreen and sweet birch oils;

benzoic acid (or benzoates) in gums such as benzoin, or in ylang ylang or cananga oils;

eugenol in the oils of clove bud, clove stem and cinnamon leaf;

methyl and *methyl anthranilate*, *cinnamic aldehyde*, *vanillin* in vanilla.

Many other aromatic organic compounds can also be determined in the UV range; these include di-etc.

B. VISIBLE

Absorption spectrophotometry in the visible range (380 to 800 m μ) offers many possibilities for the essential oil chemist. The advantage of colorimetric methods is the extreme sensitivity; in many cases, the disadvantage is lack of specificity.

In addition to the many color-complex reactions recorded for aldehydes, ketones, alcohols, etc., direct color analyses can be carried out on products such as the oleoresins of paprika, capsicum, vanilla, turmeric and on extracts and concentrates of colored fruits. In the analysis of oleoresin paprika, simultaneous nesslerimeter and colorimeter values for the same material have been run, the values compared, and, by use of an empirical constant, the colorimeter value can be converted to the more generally accepted nesslerimeter units.

Visible methods can be used to examine raw material before processing. By dissolving a colored component from a finely ground root or fruit, the maximum yield from a particular lot can be estimated. This will determine the efficiency of various factory production processes in the isolation of the available colored component in the finished product.

Colorimetric methods for citral in lemon or lemon-grease oils are in use (Agricultural and Food Chemistry, November 1958, pages 858-60).

C. NEAR INFRA RED

The use of the near infra red range (800 to 3,000 m μ) has only recently become practical because of the development of new instrumentation some four years ago. The introduction of a tungsten lamp source, quartz optics, and a lead sulfide detector gives good results and has established the importance of the instrument to our industry. Since only hydrogen stretching bonds and their overtones absorb in this range, the usefulness of the instrument is somewhat limited. However, the near infra red range is often applicable in certain cases where other instrumental methods give vague or uncertain results.

When a solvent is required, Carbon Tetrachloride is usually employed. Using a one centimeter cell, a 5% solution of the sample is recommended for the range above 1.6 μ ; below 1.6 μ , an undiluted sample can be used. It is well to have several cells of different thickness available; 0.1 cm., 1 cm. and 10 cm. cells are in general use.

Hydrogen atoms bonded with carbon, oxygen or nitrogen exhibit strong peaks in this range; any environmental effects will also be apparent. Thus, near infra red can differentiate the hydrogen atoms in methyl, methylene, vinyl, vinylidene, or acetylenic groups; also it will indicate whether the hydrogen is attached to a ring (such as in aromatic, mono and bicyclic compounds).

Aliphatic aldehydes can be distinguished from aromatic aldehydes. The hydroxyl group of glycols, phenols and alcohols exhibit different peaks. Formates, acetates and higher fatty acid esters show specific bands. Esters of conjugated unsaturated acids are readily apparent; also the aromatic esters and lactones.

Moisture can rarely be tolerated in NIR because of its strong absorption. This absorption, however, can be used for the determination of water in solvents and other products.

For factory control, NIR is useful in following the reduction of aldehydes to alcohols and in determining the amount of unreacted alcohol in acetal or ester formation.

For quality control of essential oils, NIR can be

useful. However, it is difficult to make complete band assignments, but by using specific bands (such as those mentioned above) much valuable information can be obtained.

D. INFRARED

Among the first organics investigated by IR were several essential oils examined by Donath in 1896. Today, the IR recording spectrophotometer has become the most valuable instrument in the essential oil instrumental laboratory. Much IR data have been published dealing with essential oils and the related products of our industry. Since most of these papers are readily available and are familiar to the essential oil chemist, we shall limit our discussion to the general application of infra red techniques.

The use of infra red for evaluation and quality control of essential oils is well established. However, it should be remembered that in preparing standard reference curves, it is of utmost importance to use only oils whose purity and authenticity is beyond question.

In the United States, the use of infra red for factory control has become extremely important. All reactions which involve a functional group of any type can be followed by the use of such instruments. Reactions such as ester formation, reductions, oxidations, condensations, substitutions, additions, dehydrations, hydrations and isomerizations all lend themselves easily to quantitative determination by infra red. Also, the selection of raw material for manufacturing operations is often made on the basis of IR examination.

Research on the constituents of essential oils has progressed rapidly because of IR. Many constituents have been isolated by wet methods, column chromatography or preparative gas chromatography and then identified or, if unknown, their structures determined. For such work, a file of pure reference curves is a necessity; many laboratories have been forced to use IBM (or other punch card systems) for filing of the complex and multitudinous IR information.

In many cases, the main failing of the IR method is its lack of sensitivity to trace components. This particular problem can sometimes be overcome by proper sample preparation; fractionation (or other type of separation) will increase the concentration of trace material in the sample under investigation. Recent instrumental improvements have also helped to solve this problem. Micro cells, beam condensers and scale expanders are now available. The small samples resulting from VPC separations have made micro sampling techniques necessary.

VAPOR PHASE CHROMATOGRAPHY

The most recent instrumental advance has been VPC or Gas Chromatography. Known for several years, it was applied to the separation of hydrocarbon isomers by the petroleum industry with excellent results. Instrument companies in America quickly produced a commercial instrument for general application and the technique was applied to all phases of organic chemistry. The unique ability of this instrument to separate organic compounds, even isomers and homologs, was reported by workers in many fields. The instrument seemed especially adapt-

able to complex mixtures such as essential oils. The larger essential oil companies were soon building their own instruments or modifying commercial instruments to fit their needs. Today, there are about 15 instrument companies manufacturing some 30 to 40 different models of VPC equipment. For particular applications, instruments are available combining various modifications of detectors, column shapes and designs, ovens and temperature controls, sampling systems, sample recovery methods, integrators and recorders.

We must remember, however, that in all these elaborate instruments, the actual separation takes place in the column itself. Much discussion and effort have gone into the study of proper column preparation. Column material is often critical. Glass (or stainless steel) is preferred, although aluminum is sometimes used. Monel and copper may cause difficulties in the presence of certain compounds as, for example, phenols. Column packing is also very important; it must be inert. Celite, firebrick and other materials contain iron salts which are undesirable since they are excellent isomerization catalysts. Although the slightly acidic nature of these products may not interfere with fatty acid separations, it will dehydrate citral to p-cymene or alpha terpineol to dipentene. Hence, for use with essential oils, it is best to acid wash (conc. HCl) the inert packing, wash with water, neutralize with 1% alcoholic KOH or Na_2CO_3 and dry, leaving a slightly alkaline material. The liquid substrate, dissolved in a suitable solvent, is introduced and the final material sieved to produce proper flow rate (from 50-100 cc per min.). The mesh size employed is usually 60-80 (or 60-100). The column is then packed firmly and evenly. Materials that have been proven to be excellent separators for essential oils are:

UCONS—polyalkylene glycols and their aliphatic diesters;

CARBOWAXES—polyethylene glycols;

"TIDE"—a detergent—alkyl aryl sulfonates;

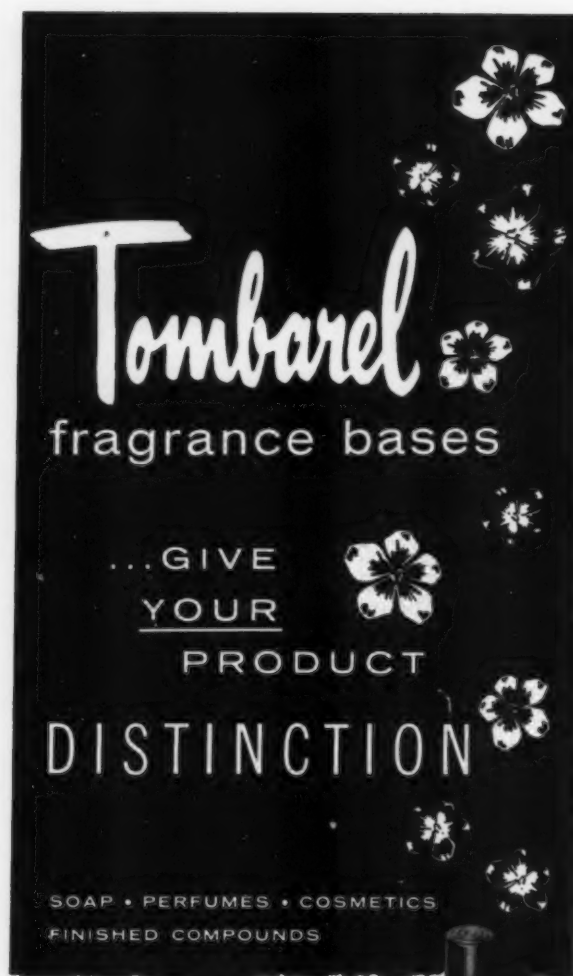
APIEZON WAXES

SILICONE OILS

Vertical columns give best results; coiled columns use less oven space and, where the radius of the coil is at least ten times the diameter of the column, excellent results can also be achieved. Tighter coils will not separate properly.

Hot wire detectors, slightly more sensitive at temperatures above 155°, cause carbonization of organic materials in essential oils, quickly become dirty and must be replaced. Thermistor beads, less sensitive at higher temperatures, do have stability and longer life. The new beta-ray, flame-ionization or radio frequency are extremely sensitive. This sensitivity was much sought after, but, now achieved, has its drawbacks. Difficulties exist with sample reproducibility of the micro amounts which these detectors require. Too many peaks appear which are extremely difficult to identify. Some essential oil companies are using ionization detectors and capillary or Golay columns, the ultimate combination for sensitivity for trace constituents; no results as yet have been published.

We should mention here preparative columns, which are large diameter columns $\frac{1}{2}$ inch or



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more) used to separate sufficient volumes of sample for identification procedures. In a $\frac{1}{2}$ inch column, 1 to 2 ml. of a sample can be introduced. The effluent gas is passed through a cold trap, thus condensing and recovering sufficient sample for an IR or UV curve, or for determination of Refractive Index.

The VPC has replaced most of the other instrumental methods for quantitative analysis. It is very specific, sensitive and reproducible. Any component of an essential oil, once identified, can be determined.

This is a very new field of instrumentation; it is evident that VPC offers more opportunities than any previous technique for essential oil research and analysis.

3. OTHER INSTRUMENTS

A. *Mass Spectrometry* is another instrumental method finding wider use. Its ability to establish structural relationships on pure vaporizable materials has solved many problems otherwise unapproachable. Since it is founded on measurement of the masses of ionized fragments of each chemical entity, the number and variety of possible fragments from a complex mixture, such as an essential oil are very great, making interpretation extremely difficult. Its use on isolates or fractions or in conjunction with VPC isolates, however, now appears very promising. Unfortunately, the cost of the instrument is very high.

B. *Nuclear Magnetic Resonance* must also be applied to pure materials. Carbon 12 or Oxygen 16 do not respond to this technique because of equal number neutrons and protons. Carbon 14 and Oxygen 17 will react, however, and therefore some carbon and oxygen functions can be measured. The environmental effects on hydrogen in organic compounds is very pronounced and, upon this principle, investigation of essential oil isolates can proceed. The method is especially sensitive to hydrogen in methyl, methylene, vinyl, vinylidene, tertiary hydrogen, hydrogen attached to rings or on acids or esters. The solvents for samples are limited to such things as deuterated materials, or carbon tetrachloride, cyclohexane and benzene. The possibilities of this instrument are very great.

SUMMARY

In the United States of America, instrumental analysis of essential oils and related products is widely used, not only for quality control, but also for research, development, factory control and selection of raw material for manufacture.

Spectral methods, including the ultra-violet, visible, near infra red and infra red ranges are commonly used. These methods are well established and accepted.

At the present time, vapor phase chromatography is being enthusiastically received. Most of the intensive work today centers about this instrumental approach. Much can be expected of it in the near future.

Mass spectrometry and nuclear magnetic resonance are relatively new to our field. Very little work has been carried out with these instruments. Whether or not they will take their place with the established instruments remains a question for the future.

Emulsions in Theory and Practice

A report on a joint meeting of the Surface Activity and the Oils and Fats Groups of the Society of Chemical Industry in London on January 11th, 1960. Dr. C. G. Sumner, M.Sc., Ph.D., F.R.I.C. of the British Standards Institution gave a paper titled "Emulsions in Theory and Practice". This report gives a summary of this paper, together with some comments made by others at the meeting, which was attended by about 80 persons. This report is through the courtesy of the Hon. Recorder F. Riley.

Dr. Sumner discussed the present indiscriminate application of the term 'emulsion' to systems made by emulsification, irrespective of whether or not both phases were liquid in the final product. He then outlined the development of emulsion technology, and pointed out that the theoretical advances which had had the most impact on technical progress were all concerned with emulsifying agents. Outstanding among these was the development of synthetic surface-active agents as a result of the theory of molecular orientation at interfaces.

Because of this historical background, emulsion theory had become concentrated on the properties of the interfacial film as such, and the influence of the film on the mutual relations between film-covered droplets in an emulsion had received comparatively little attention. There were three main problems to be considered in this connection:

- (1) How the nature of the emulsifying film determined the phase relationship in the emulsion.
- (2) How the film enabled the droplets to persist without coalescence.
- (3) How the film influenced the bulk properties of the emulsion, e.g., the state of aggregation and the flow properties.

Phase relationship was due to the selective action of the film in opposing the recoalescence of droplets of one liquid but not of the other; the mechanism of this selective action was still uncertain. Persistence of the droplets after formation of the emulsion was an allied problem, and it was not yet known how far this depended on the maintenance of a layer of continuous phase between droplets even on close approach. Flocculation in oil-in-water emulsions could occur if the concentration of emulsifying agent was either too low or too high, or if the agent was insufficiently hydrophilic when adsorbed at the interface. The viscosity of the emulsion was increased by flocculation, but another possible factor in the flow of concentrated emulsions was the effect of the interfacial viscosity or rigidity of the film in opposing distortion of the droplets.

The Chairman of the Surface Activity Group Sir Eric Rideal, M.B.E., F.R.S., who was Chairman of

the Meeting, opened the discussion on Dr. Sumner's paper and the following points were raised:

Dr. T. Malkin

Dr. Sumner has referred to Dr. Lawrence's remarks on how little we know about emulsions. Of course, we know a good deal, but the variables are so great that it is unlikely that any simple theory will embrace all the phenomena. If we consider for example the coalescence of two oil drops in an oil/water emulsion, they must first overcome the electrical double layer and make contact, then as the point of contact broadens into an area, the surface active agent at the junction of the drops must move either into the oil or into the aqueous phase as the original two surfaces merge. The facility of this process will depend on the size, shape, and character of the hydrophobic group and the polarity, shape and hydrogen bonding power of the hydrophilic group. The precise part played by each is a difficult problem in itself.

Dr. Sumner

The fact that the coalescence of droplets in an emulsion is a complicated phenomenon has been insufficiently realised in the past, and one of my main objects in the paper has been to stimulate interest in this and other neglected aspects of emulsion behaviour.

Mr. E. M. Learmonth

Dr. Sumner's discussion seemed to imply that the oriented molecules at the interface were static and even bound in a rigid structure. He spoke, for example, of "rupture of film". This picture was rather differently painted by the Chairman some years ago, discussing the orientation of grease films in connection with detergency. There it seemed that the oriented molecules might individually reverse their orientation and the frequency with which this occurred was a function of their free energy. Is not this a more likely picture, and could it not be utilised to classify at least the simpler types of long chain molecules whose energy characteristics, especially in homologous series, are perhaps known by now in fields quite outside that of emulsion technology.

If these speculations have any basis one might be able to predict the relative likelihood of coalescence of droplets stabilised by members of homologous series and extend the data to bring in other factors such as the degree of solvation of polar groups and so forth. Is sufficient data available for this sort of consideration?

From Dr. Sumner's discussion of protein stabilisers too, I gained the impression of a rigid mechanical structure. Is it not possible here also that a degree of movement is occurring all the time? At least in the aqueous phase one might suppose protein molecules to be capable of a good deal of relative motion, related for example to their degree of hydration and to the varying strength of their ionic charges on which data is surely accumulating by now.

Dr. Sumner

That the molecules in interfacial films are not stat-

ic is generally recognised, but is hardly likely to affect the arguments discussed in the paper. The kinetic approach suggested by Mr. Learmonth does not differ fundamentally from the calculation of HLB numbers by J. T. Davies. My reference to rigid protein films was to the condensed, three-dimensional thick films which are readily built up from protein monolayers, and did not imply that the individual molecules are static.

Dr. R. H. Marriott

The presence of a "third" layer of materials at the interface between two immiscible liquids which have been emulsified, is of considerable interest.

Experimental work has been carried out in our laboratory to measure the dimensions of this film and the evidence so far obtained in the case of an emulsion of water-in-oil made with white oil, beeswax and a milk alkali suggests that this third state behaves as though it were somewhat crystalline (or orientated) and is of the order of eight molecule lengths in thickness. Bearing in mind the molecular length of the esters in beeswax, this is of significant magnitude. Our view is that the presence of such a boundary virtually increases the volume of the dispersed phase so that the emulsion possesses properties which one can look on as pseudo-thixotropic.

That this boundary has rigidity can be shown under the microscope by allowing a tiny drop of the emulsion to dry slowly in air, observing the phenomenon at the edge of the droplet. The water globules are seen to begin to collapse but not in the fashion that one would expect if the carapace had rubber-like properties. The water appears to be lost as vapour through the thin layer of the surrounding oil and the globules collapse in a similar fashion to a table-tennis ball if it be crushed by external pressure. The remnants of the rigid film can just be seen under the microscope, when the whole of the water has disappeared, as an indefinitely-shaped residue.

Dr. Sumner

Some work on wax dispersions which I carried out many years ago indicated that the emulsifying agent formed by beeswax and alkali is different in character from an ordinary soap, and the experiments described by Dr. Marriott are of great interest. The transfer of liquid from a droplet by molecular diffusion through the emulsifying film is doubtless a factor to be taken into account in the coalescence of droplets.

Mr. E. D. Gilbert

There is a tendency among workers in emulsion chemistry to treat the interfacial film as if it were a fixed, unchangeable structure, once it is formed. This is wrong. The interfacial film is in equilibrium with both phases, and changes in the composition of either phase will modify its structure, and so affect the stability of an emulsion. The effect of temperature on emulsion stability can be explained in the same way. Simple experiments in the solubilisation of kerosine in a dilute solution of a secondary alkyl sulphate will illustrate this very clearly. Experimental details

are published in my paper in Proceedings of Second International Congress on Surface Activity Volume 1.

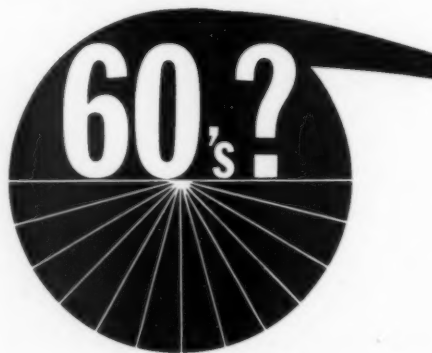
It is also demonstrated clearly in these experiments how addition of electrolytes (e.g. sodium chloride) can improve the stability of emulsion which is contrary to the generally accepted belief that electrolytes invariably cause instability. In fact it is sometimes practicable (and moreover economical) to use salts to improve the stability of emulsions based on castor oil soaps.

One of the great difficulties confronting the worker in this field is the lack of precision in the terms used to describe phenomena. For example, the word inversion has been used to describe (a) phase inversion without separation, (b) phase inversion with partial separation of one phase and (c) breakdown of the emulsion into two phases. The Surface Activity Group of the Society of Chemical Industry could make a great contribution to the study of emulsions by helping to introduce some precision into the terminology.

Dr. Sumner

It is useful to emphasise that the nature of both the liquid phases influences the properties of the interfacial film. On the need for more precise terminology I strongly agree; as pointed out in the paper, even the term 'emulsion' itself is used ambiguously, and it would be helpful to have a new term to describe a system which is made by emulsification but is not necessarily an emulsion in the classical sense under the conditions of use.

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BRANCHES THROUGHOUT THE WORLD



Progress in perfumery materials

BY PAUL BEDOUKIAN

WITH THE YEAR 1960, we are undoubtedly entering the space decade—as well as the aerosol decade—and efforts to keep ahead of our friends and enemies continue at a furious pace.

In many important areas of chemistry, we are maintaining our leadership and making important contributions of benefit to mankind. In our field, however, there is little fundamental research being done in this country. Most of our larger companies are apparently concentrating on improving the quality of their products and lowering costs. If this trend persists, it is likely that any major developments will continue to come from sources other than the aromatics industry.

Pure research in aromatics is being carried out in Europe and the Soviet countries, so that we can look forward to new products and processes useful in our industry.

As a result of a major breakthrough in the manufacture of synthetic linalool and related materials, much effort has been directed toward making the whole range of terpenes synthetically. Many ingenious methods have been tried and some of these will no doubt be commercially successful. A number of these reports are mentioned in this article.

A major breakthrough was also made in compounds possessing an ambergris odor. There are relatively high molecular compounds which possess unbelievably powerful odors and they may well influence our thinking on the perception of odor.

Alongside the important contributions of the chemist to the aromatics industry, are the continuing efforts of the essential oil producer to improve the yield and quality of oils. With the demands made by an increasing population and rising costs, he seems to be fighting a losing battle, but his produce is still indispensable as essential oils in large quantities are still used in most formulations. The production of essential oils in the so-called underdeveloped countries has become quite important.

As in previous years, much interesting material has

been left out of this article in order to keep it within the scope of this publication. Only articles of general interest have been included and the writer regrets the necessity of omitting many valuable reports dealing with fundamental problems of terpene chemistry.

Odor and its Perception A great deal of experimentation has taken place to further our understanding of odor and its perception, but the problem remains essentially unsolved. A paper on smell compensation written fifty years ago by Zwaardemaker and recently translated in a British journal, reads very much like an article written today (1). Continuing his earlier work, Lauffer (2) has made an exhaustive study of the subject and has again ably reviewed the accumulated data. This paper, originally appearing in the Toilet Goods Association publication, has been reprinted in British (3) and American (4) trade journals.

A lengthy philosophical discussion under the title of "Sympathy and Odour" has been translated from the German and appeared in a British perfumery journal (5). Another paper points out some interesting factors concerning odor and its perception (6). The detection and correction of unwanted odors in various packaging materials and commercial products have been discussed in a recent publication (7).

Perfumes. Perfumery is an art which calls for the



Paul Bedoukian, Ph.D., the author of this article, is a consulting perfume chemist, and author of *Perfumery Synthetics and Isolates*. This review of developments in this field is his sixteenth annual effort, and together with the previous reviews, provides a truly encyclopedic collection of all pertinent information and literature in the field of perfumery.



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creation of compositions having desirable odor characteristics. It is inevitable that the perfumer does not always agree with the analytical chemist as to methods of judging and controlling perfumery raw materials (8). Some of the factors involved in creating compositions for perfuming various mediums are considered in an interesting article, "Physico-Chemical Behavior of Perfume Materials in Various Carriers" (9).

Certain plastics have been proposed as being suitable containers for liquid fragrances. An excellent study lists the behavior of a wide range of perfumery materials when stored in polyethylene containers (10). Another article discusses the use of aldehydes and some acetals in perfume formulations (11).

Maurer is continuing his interesting series of articles on the odors of various families of plants. Recent publications include discussions on the odor qualities of the geranium rose, fuchsia and saxifragaceae families (12).

Fixation of perfumes is an important factor which must be considered when developing or creating formulations. It is reported that musk sage markedly decreases the volatility of perfumes. The author concludes that the lasting quality of a perfume is related to its volatility with steam (13). A German patent claims that silicone oil with a suitable viscosity is a good fixing agent for perfumes (14). Another German patent describes a solid solution of a perfume using urea-formaldehyde resin as the solvent phase (15).

A Soviet patent describes an apparatus which clarifies liquid fragrances electromagnetically (16). There is an interesting article on the use of ion exchange resins in the perfume industry (17).

Flower and Other Oils. Substantial amounts of flower oils are still essential for the creation of really high-class perfumes. We do not have as yet a truly satisfactory substitute for fine flower oils.

Rose and jasmine are undoubtedly the most important flower oils used in perfumery and it is not surprising that they are subjected to so much study. It is reported that the quality of oil is the same whether obtained from the petals or the whole flower by steam distillation (18). An 80-100% increase in the yield of rose oil was reported, by first fermenting the petals of roses under controlled conditions (19). The hydrocarbon stearoptenes of Bulgarian rose oil have been studied (20) and their composition noted (21). Paper chromatographic studies have shown the presence of nonyl ethyl, methyl and phenyl ethyl alcohols in Bulgarian rose oils (22).

The oil obtained from *Rosa centifolia* grown in Calabria has been subjected to partition gas chromatography and shown to contain the following constituents: nonyl aldehyde, linalool, phenyl ethyl alcohol, rhodinol, nerol, geraniol, geranyl acetate, eugenol, and possibly methyl eugenol. Propionic aldehyde, isovaleraldehyde, phenylacetaldehyde, salicyl aldehyde and citral were also detected (23).

In studying the cultivation of jasmine oil in Egypt, it was found that essential oils are formed during the blooming stage of the flowers, whereas the waxes

are formed before blooming. The highest yields and best oils were obtained during the half-blooming or artificially bloomed stages, both activated with hydrochloric acid. An increase in the yield of oil was obtained by freezing the flowers before extraction of the perfume oil (24). Russian workers reported a four-fold increase in the yield of jasmine flower oils by blowing moist air through the flowers and extracting the volatilized perfume oil on activated charcoal (25).

Seventy-five kilograms of lily (*Lilium auratum*) petals were extracted with ether and 6-7 grams of oil were obtained from the concrete. Among the compounds reported were vanillin and p-hydroxy-methoxytoluene (26) detected by classical methods of analysis. A similar study was made on chrysanthemum flowers and the presence of anisaldehyde, bornenol and its acetate noted (27). A major component was 2,2,4-trimethylcyclo-hex-3-ene-1-carboxylic acid. Methods of extracting oil from azalea flowers (28) and its possible use in perfumery (29) were reported by Russian workers.

Steam distillation of mulberry leaves gave an oil containing among other constituents, beta-gamma hexenol, secondary octyl, benzyl and phenylethyl alcohol (30).

Extraction of fresh and fermented strawberry juice gave an oil which was then studied. The presence of some hitherto undiscovered compounds was reported (31).

A lengthy article enumerates the odors of products of animal and plant marine life. Some of these contain nitrogen and sulfur compounds. Algae contain various low molecular acids and a range of terpene compounds (32).

Japanese workers have been spending a great deal of time in the study of essential oils obtained from tobacco leaves. No less than sixty-five components were identified in this oil covering a wide range of chemical compounds (33). The tobacco industry has long been a user of various perfumes and flavors. A recent patent reports that the addition of small quantities of gamma isobutyl gamma butyrolactone added to tobacco greatly improves its aroma and smoke flavor (34). A new cyclic ketone, 4-(3-ethyl-2,6,6-trimethyl-2-cyclohexen-1-yl)-3-buten-2-one is reported to be an aromatic having the fragrance of fine Havana tobacco, and as such is useful as a tobacco perfume (35).

Analytical Procedures in the Perfume Industry. Vapor phase chromatographic techniques are the new rage and like all newly developed methods are receiving much attention and publicity. One hears very little about some of the difficulties encountered in the use of this technique or of the pitfalls awaiting the untrained analyst. No doubt it will eventually be accepted as a useful tool in certain types of analyses of perfumery materials.

The analysis of bergamot and lemon oils by gas chromatography is described by two Italian workers (36). Its general application to the perfume industry is described in a brief paper (37).

A refinement of vapor phase chromatography is gas-liquid partition phase chromatography. The comparative merits of the Glichitch and Fiore methods of

determination of linalool have been studied by gas-liquid partition chromatographic analysis. It was found that the Fiore method gave values about 1% lower and the Glichitch methods about 4% lower (38).

Using standardized techniques, it is possible to employ vapor phase chromatography for the general analysis of essential oils and it is claimed that the results obtained for certain mixtures are within 0.5% of theory. Based on these results, partition gas chromatography is recommended as an official method of examination of essential oils (39). The analysis of such oils as eucalyptus, cajeput, etc., was reported to give results with an error less than 5% and the method therefore offers definite advantages over older procedures (40). Thyme oils gave results which are claimed to be more accurate than the standard alkali extraction methods (41). Eugenol in essential oils is determined with an accuracy of 2 per cent by this method (42). Other workers have pointed out some uses of vapor phase chromatography in the analysis and examination of essential oils (43) (44).

According to a recent report, racemic compounds may be resolved by vapor phase chromatography, using an optically active stationary phase (45). Further development of this technique may prove to be extremely valuable in the examination of perfumery materials.

Paper chromatography has its limitations in our industry because of the difficulty of preparing derivatives of terpene compounds. There are, however, definite areas where this very useful tool can be employed profitably. For example, menthol can be determined in the presence of thymol and camphor by this technique. The xanthates are separated and the color produced with p-aminobenzaldehyde (46). The lower fatty acids occurring in lavender oil were successfully detected by this method (47). A Japanese paper points out the usefulness of paper chromatography and the additional advantages of paper electrophoresis in the separation of the components of essential oils (48).

The value of infra-red spectroscopy as an adjunct to separation techniques is brought out in an interesting publication (49). Its general application to the essential oil industry is indicated in an Italian publication (50).

Raman spectra can be used in the analysis of essential oils or their fractions (51).

In analyzing perfumes and colognes, it is often necessary to determine their alcohol and water content. It was reported that alcohol can be determined with dichromate and water by measurement of the ammonia formed after reaction with Mg_3N_2 , and the essential oils, by binding with dibutyl phthalate (52). Water can also be determined by the Fisher method, and alcohol by acetylation (53).

It is always difficult to carry out a determination of carbonyls in dark colored oils. This difficulty can be overcome by using a potentiometric method of estimation of the formation of derivatives, as described in a recent paper (54).

New Approaches to Synthetic Terpenes. It is indeed fascinating to review the different approaches toward synthesizing terpenoid perfume materials.



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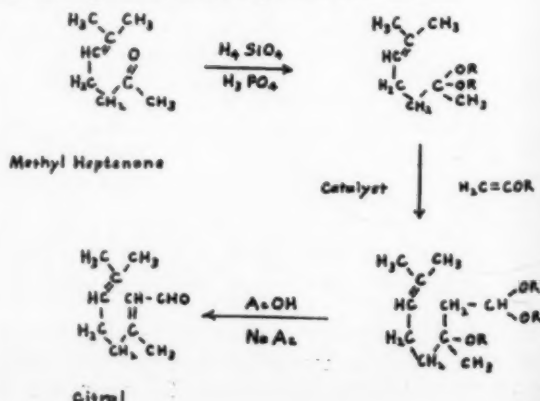
One basic building block which is readily available is isoprene or its derivative. It would be highly desirable to couple and hydrate isoprene to obtain terpene alcohols, but this is not readily achieved and more direct approaches are necessary.

Citral has been produced by adding hydrogen chloride to isoprene and subjecting it to a series of reactions, as pointed out in our last annual review (54). This synthetic citral has been compared to the natural product in purity and proportions of structural isomers (55). The reaction of hydrogen chloride addition to isoprene, followed by telomerization to produce geranyl chloride was subjected to a critical study by Soviet chemists (56). Conversion of geranyl or methyl geranyl chloride to citral and methyl citral was also described (56a). A Soviet patent discloses the process for the condensation of isoprene chloride with acetoacetic ester to give methyl heptenone (57).

Instead of using isoprene chloride, it is possible to start with methyl butenol ($\text{Me}_2\text{C}(\text{OH})\text{CH}:\text{CH}_2$) towards the synthesis of terpene products. It readily forms the bromide which can be reacted with acetoacetic ester to give methyl heptenone and then dehydrolinalool through the well known reaction. Further condensations give egranyl acetone, farnesol, farnesyl acetone and other valuable products. A report on a study of these reactions appeared in a Soviet chemical journal (58). Methyl butenol can be condensed directly with acetoacetic ester to give methyl heptenone which is then used to obtain the series of compounds mentioned above, as pointed out in a French publication (59). Polymerization of methyl butenol with potassium bisulfate gave a range of isomeric terpenes, including geraniol, linalool, geranyl hydrate, etc. (59a).

Since methyl heptenone is so readily available, the next step, namely, its condensation with acetylene to give dehydrolinalool, becomes of prime importance. This step is described in an American patent which includes the one-step hydrogenation of the acetylenic compound with sodium in ammonia to give linalool (60). A German patent describes the condensation of acetylene with methyl heptenone in tetrahydrofuran using sodium as the condensing agent (61).

A rather unusual reaction was reported in a Soviet patent. The ketal of methyl heptenone is condensed with vinylates to give the 1,1,3-trialkoxy derivative, which on heating with acetic acid gives citral (62) according to the following series of reactions.



This reaction has been applied to other olefinic compounds (63) (63a).

It may be recalled that the pyrolysis of acetoacetic esters of allylic alcohols gave gamma olefinic ketones and thus led to the practical synthesis of ionone. The pyrolysis of the acetoacetates of enynic alcohols led to the formation of enynic ketones and no polyene ketones were formed according to a recent study (4).

Although the reactions appear to be controllable when starting with isoprene, one may also attempt to start the synthesis of terpenes with the readily available hydrocarbon myrcene. Unfortunately, it is not yet possible to hydrate myrcene directly to linalool, and so far, judging from literature reports, synthetic terpene alcohols are made from myrcene by first adding halogen chloride followed by hydrolysis. We thus find several American patents describing such processes. One patent discloses the addition of HCl to myrcene followed by hydrolysis of the chloride under mild conditions to give linalool, terpineol, geraniol, and other products. A second patent describes the treatment of the myrcene chloride with sodium hydroxide and Triton X-100 to give alloocimeneol possessing a rose and lily of the valley odor (66). A third American patent reveals the fact that myrcene hydrochloride consists of a mixture of chlorides which on hydrolysis give a mixture of linalool, terpineol and myrcenol (67). Apparently, better controllable yields of alcohol are obtained by first hydrogenating myrcene to dihydromyrcene and then adding hydrogen chloride to give dihydrolinalyl chloride. This, on hydration, gives dihydrolinalool, according to a recent American patent (68).

Another method of obtaining alcohols from hydrocarbons is to form the epoxide of the hydrocarbon and then hydrogenate it to the alcohol. Dimethyl octanol was thus obtained from the epoxide of tetrahydromyrcene (69). Hydrogenation of limonene epoxide yielded isocarvomenthol, and of pinene epoxide, isopenocamphenol (70). Other workers reported the formation of carveol and dihydrocarvone by boiling limonene epoxide with alumina. Carveol acetate and the enol acetate of dihydrocarvone were obtained by boiling the epoxide with acetic anhydride (71).

A novel method of hydration of terpene hydrocarbons involves the use of diborane (72).

According to an American patent, alpha pinene yields a wide range of valuable oxygenated compounds when air is blown through it to give a measured amount of peroxide which is then reduced in peroxide value by treatment with alkali. The resultant product is fractionally distilled to give a range of valuable oxygenated terpene compounds (73).

A new method of synthesizing alpha beta unsaturated aldehydes, such as citral, was described in a recent publication. It involves a rather novel rearrangement of ethynyl carbonyl acetates to give the acetals of the required aldehydes according to the following scheme (74):

Terpenes. Optimum conditions for the extraction of geraniol from palmarosa oil by means of calcium chloride addition product have been thoroughly investigated (75). Citral is often more readily available than geraniol and it is possible to selectively hydrogenate it to a mixture of nerol and geraniol (76). A study of citral and related compounds appeared in a recent publication (77).

By controlling dehydrogenation conditions, it is possible to convert geraniol to either citronellal or citral (78). Such conversions have found industrial application in the past, and have recently been described in a Soviet patent (79).

Since the absolute configuration of citronellal and pulegone was known, it was possible to determine the absolute configuration of citronellic acid and its methyl ester and amide, of citronellol and its bromide, and the hydrocarbon derived from it (80).

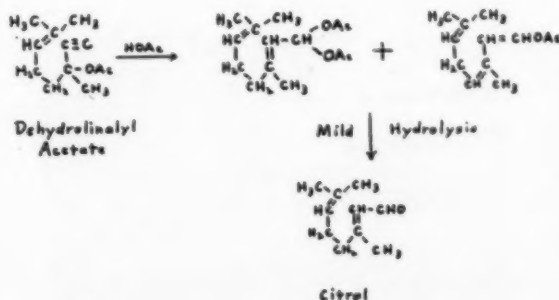
A study was made of the various factors affecting the yield of hydroxycitronellal from citronellal, using standard manufacturing procedures (81). An optimum yield of 65 per cent was claimed in this paper. The accepted structure of hydroxycitronellal was shown to be correct in another report (82).

Linalool suitable for perfumery purposes was obtained by careful fractional distillation of coriander oil (83) and muscatel sage oils (84 grown in the Soviet Union).

Some of the products formed by boiling linalool with acetic anhydride for pre-determined periods were studied by means of vapor phase chromatography. Formation of the unsaturated hydrocarbons, myrcene, ocimene and limonene, as well as geranyl acetate, was reported (85).

The manufacture of terpineol from terpin or terpin hydrate was disclosed in a German patent (86). A recent paper reports the synthesis of cis and trans beta terpineol (87). A new crystalline terpineol, obtained from the oil of citrus unshiu was reported to have properties differing from the known alpha, beta or gamma terpineols (88).

A method has been developed for manufacturing menthol from pinene, and was disclosed in a recent American patent (89). Since levo menthol is the most highly prized isomer, efforts are being made constantly to convert other isomers to either the levo or a mixture of dextro and levo forms. Thus, two recent American patents describe methods of converting dextro and other racemic menthols into a mixture of dl-menthol by heating the mixtures in the





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presence of hydrogen and catalysts (90) (91). Corn-mint oil is still an important source of menthol. A Japanese patent describes the use of nitromethane as a solvent for the extraction of menthol from mentha oils at low temperatures (92).

Causes of the presence of menthofuran in essential oils in labiates from Albania and northern Greece, are discussed in a review (93). This troublesome compound has been subjected to repeated studies. It has recently been prepared from the epoxide of isopulegone by treatment with benzoyl peroxide (94). The flavor chemists find menthofuran undesirable because of its tendency to oxidize to compounds having undesirable flavor characteristics. In this connection, it is of interest to note a study which has been made for the purpose of determining the structure of oxidation products of menthofuran (95).

dl-Carvone has been obtained by pyrolysis of 8-acetoxycarvotanacetone formed by the oxidation of terpinyl acetate (96) (97). Thujone was obtained in very high yields by oxidation of thujol which is made by hydrogenating sabinol obtained from juniper oil (98). Isobornyl formate is manufactured continuously by treating camphene and formic acid with ion exchange resins (99). The reaction products of pinene and acetic anhydride were subjected to a thorough study. The products obtained include: campholenaldehyde, sobreril acetate, trans carveol acetate, trans carveol, etc. Similar studies have been made with carene and its reaction products with acetic anhydride (100).

Two university publications deal with the chemistry of the eucarvone series (101) (102).

Other investigations include the preparation of perillyl acetate by treatment of nopinene oxide with acetic anhydride, followed by pyrolysis (103), the oxidation of carene to yield a new carene alcohol (104), and the preparation of isopiperitone and trans-piperitol (105).

Research in Musk Compounds. Macrocyclic compounds having musk like odors are valuable for perfumery purposes and they also present challenging problems to the organic chemist.

A production-feasible synthesis of macrocyclic lactones by cyclizing omega hydroxylic acids was reported in a lengthy paper (106). The preparation of lactones having 15, 16 and 17 carbon atoms was reported by these workers. A recent Soviet patent disclosed the preparation of macrocyclic lactones and oxalactones from tetrachloroalkanes. A Japanese paper described the synthesis of cyclopentadecanone using undecylenic acid as the starting material (108).

These macrocyclic compounds are formed by cyclization of the corresponding acids. In this connection, it is interesting to note an Indian patent describing the manufacture of civetoe-dicarboxylic acid (109) from oleic acid. Electrolysis of a mixture of omega acetoxundecanoic acid and monoethyl adipate is reported to give 1 per cent omega acetoxundecanoate which can be used for the preparation of cyclopentadecanolide (110).

The reported preparation of macrocyclic acetylenic compounds (111) and macrocyclic polyenes (112) are of academic interest and worthy of mention.

This report will be concluded in the April issue.

Dihydroxyacetone "sun tan"

by LEONARD BARMAN, Commercial Development, Wallerstein Company

A "tame bug" and a couple of knowledgeable scientists have brought Florida suntans into the homes of winterbound northerners. A species of *Acetobacter* was taken in hand by Drs. Theodore Cayle and Samuel Green of the Wallerstein Company who fed it a diet of glycerol. Result? Cayle and Green came up with a method of producing dihydroxyacetone in quantity at quantity prices. Now selling at \$7.94 per pound (the old price used to be about \$2.00 a gram), this new fermentation chemical is the "secret ingredient" of lotions that create a synthetic suntan on the user's skin.

The skin coloring mechanism is thought to proceed through the reaction of dihydroxyacetone with the amino acids of keratin, such as tyrosine, phenylalanine and tryptophan. What the mechanics of the reaction may be is unknown, for dihydroxyacetone is an extremely reactive molecule—the carbonyl group, hydroxyls and methylol hydrogens all being reactive enough to participate in some sort of coloring reactions.

Other reactions of dihydroxyacetone include esterifications, oxidations, condensations and cyclizations. Also, because the compound is a ketotriose, it can undergo all the reactions of this type of sugar. Some nitrogenous derivatives, interestingly enough, have shown antitubercular activity. Dihydroxyacetone also finds use as a modifier of redox initiators systems for butadiene-styrene polymerizations, and as a physiologically safe humectant. Truly dihydroxyacetone is a versatile chemical!

Physically, dihydroxyacetone is a white crystalline powder, somewhat hygroscopic. Cryoscopic measurements indicate that dihydroxyacetone is a dimer. By either melting or using heat to effect solution in a solvent, the monomer can be regenerated from the dimer. Reversion to a monomer is rapid in aqueous solutions when using freshly prepared dihydroxyacetone; it slows down as the age of the dimer increases.

The solubility characteristics are dependent upon the dihydroxyacetone molecular arrangement form, monomer or dimer. The dihydroxyacetone monomer is very soluble in cold water, alcohol, ether and acetone—the dimer only slightly soluble in cold absolute

alcohol, ether and acetone. The solubility characteristics of the monomer are obtained in these solvents by warming. Both monomer and dimer are insoluble in hydrocarbons.

Dihydroxyacetone-1-phosphate is a primary intermediate in the metabolism of glycerol and carbohydrates in microorganisms, higher plants and animals. Dihydroxyacetone is more readily metabolized than glucose and also forms glycogen more readily than glucose.

Dihydroxyacetone has been used for the treatment of diabetes and hypoglycemia and as an antidote for cyanide poisoning. No toxic reactions to internal or external use of dihydroxyacetone have been reported.

The basic concept for the future use of dihydroxyacetone is that it will be used as a basic building block for compounds of interest as pharmaceuticals. Some derivatives of dihydroxyacetone, such as a pyridine carboxylic acid hydrazide has been shown to be active against *Mycobacterium tuberculosis*. Dihydroxyacetone may also be used to synthesize other pharmaceutically useful materials.

Condensation products of dihydroxyacetone and sulfonamides have been suggested for drugs and dyes as well as tanning agents and wool preservatives. Dihydroxyacetone, as a catalyst, is effective in condensation reactions of formaldehyde to hydroxyaldehydes and hydroxyketones. Dihydroxyacetone is useful in polymerizations e.g. styrene-butadiene.

The polyfunctionality of dihydroxyacetone is the property which makes this chemical interesting to nearly all industries. Dihydroxyacetone may find its greatest use in modifying starches, textiles or paper products, or as pointed out, may be utilized in the pharmaceutical field as a basic building block for some yet unknown wonder drug of tomorrow.

A Guide to Formulation—Editor

Dihydroxyacetone	up to 2.5%
Alcohol	up to 55%
Polyol	up to 2%
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PACKAGING & PROMOTION



1—Lenthieric

A line of bath requisites, Bath of Gold by Lenthieric, combines fragrance with unique and exclusive moisturizing formulae in three forms. The richest aqua-mollient concentrate, BAIN D'OR Bath Oil for Dry Skin is a fine oil, with the fragrances of orange and lemon.

The crystal bottles, slender and taper tall are designed with an oval indentation front and back to hold gracefully without danger of slipping. A carton of yellow, marbelized with streaks of white and gold, have a BAIN D'OR logo in gold on a white medallion. A gold Greek key design is embossed around the medallion and topped with a gold swan. An insert in every package traces the history of the bath and briefly describes the BAIN D'OR line.



2—Max Factor

Timed to break right at the beginning of the vacation and travel season, Max Factor is introducing "Vacation Specials" that feature five of his most popular make-ups in special sizes and at special prices. The five packaged requisites are small enough to easily fit in any suitcase or bag yet large enough for later use as well as for a lengthy travel spree.

Pan-Cake Make-Up, Pan-Stik, and Erace will come in unit cartons featuring a white, cerulean blue, and gold color scheme, with a golden monogram in a distinctive rococo design. Creme Puff will have a specially designed cover for its handy metal case in the same matching motif and color scheme, and Hi-Fi Fluid Make-Up will be in a special-size gold top bottle.



3—Christian Dior

Christian Dior's deluxe Eau de Toilette is now presented in a more elegant box with added gift appeal. The grey box is smartly accented in white and gold, and each of the three Dior fragrances is distinguished by a different color in the decorative ribbed panelling on the front of the package. "Miss Dior" has a gold and white scheme; "Diorama," grey and white, and "Diorissimo," pink and white. The Eau de Toilette, itself, is in a crystal bottle etched with the characteristic houndstooth design, and is set off with a white bow.



4—Kings Men

Kings Men has introduced a Stick Deodorant and Anti-perspirant with a fougere spice fragrance. The taper-tall, white ribbed column-like deodorant container is capped by a pedestal of vibrant red, destined to have great masculine appeal.

5—SiBON

Perfumed bath capsules have been added to the "SiBON" Bath Line, by Monico, Inc. Aimed at attracting maximum attention for "SiBON" as a whole, they feature unusual packaging, an attractive display card, and the moderate pricing of just \$1.00. There are fifteen of the turquoise ovals in each clear plastic tube and since the screening has been done in white and gold, the interplay of contrasts proves extremely effective. The stoppers done in gold.

6—Coty

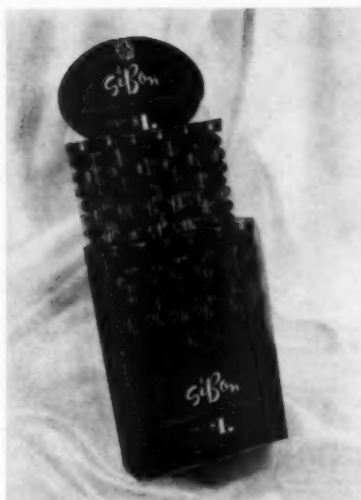
As an incentive to purchase the Coty line of fragrances a free atomizer is being offered the consumer with each \$2.00 size of Toilet Water. The atomizer, packed on top of the package in a plastic bubble, is available with any of four Coty fragrances.

7—Bourjois

Bourjois is again offering a "Spring Special" of their Evening In Paris cologne and perfume combination to retail for \$1.00. Graduating shades of blue and cerise provide the main color motif of the carton, and the windows are functional as well as decorative.

8—Mennen

Proof of purchase disks which are retained within a plastic closure are being used for the first time by the Mennen Co. on four of its cosmetic and toiletry items. These disks make it simple for the company to use in connection with promotions which call for the housewife to mail in evidence of proof of purchase. This is the first time that such disks have been used in the cosmetic industry, it is reported. Imprinted with the company's trademark, they are treated with a varnish which is odorless and tasteless, and have good resistance to water, moisture, weak acids and alkalies.



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TECHNICAL ABSTRACTS

THE COMPARATIVE PROTECTIVE EFFECTS OF DEGRADED CARRAGEENIN AND ALUMINIUM HYDROXIDE ON EXPERIMENTALLY PRODUCED PEPTIC ULCERATION, W. Anderson and F. Watt

Acute peptic ulceration of the duodenum can be produced experimentally in guinea-pigs within 24 hours of the administration of a large dose of histamine in beeswax-oil, provided a covering dose of an antihistaminic is given to protect the animals from acute shock. This method has been used to compare the effects on ulcerogenesis of degraded carrageenin (which has been shown to react with mucus lining the gastric mucosa, in low concentrations to interfere with peptic digestion, and to provide complete protection against duodenal ulceration produced by the above method) with those of aluminium hydroxide. The stomach and duodenum were removed, fixed in formol saline, and the degree of damage assessed. In the control group which received only water the maximum damage occurred in the duodenum. Considerable protection from ulceration in the duodenum was apparent in the groups treated with 10 per cent and 20 per cent degraded carrageenin, and in those treated with 2 per cent and 4 per cent aluminium hydroxide. Less protection was provided by lower concentrations of each of these drugs. In contrast, however, the degraded carrageenin afforded greater protection against stomach ulcers than aluminium hydroxide. It was observed that considerable caking of aluminium hydroxide had occurred in the stomachs of the groups receiving 4 per cent and 2 per cent gel, but this was not evident in the group receiving 1 per cent gel. In the degraded carrageenin treated groups, particularly those receiving the higher concentrations, a fine gelatinous film was observed over the mucosa, especially in the lower third of the stomach and in the duodenum. (*From the Evans Medical Research Laboratories, Speke, Liverpool, and the Department of Pathology, University of Liverpool.*) Thru THE PHARMACEUTICAL JOURNAL, October 3, 1959.

PREPARING MOLD EXTRACTS. A New Method for the Preparation of Mold Extracts Using a Synthetic Medium, Schaffer, N., Molomut, N. and Center, J. Ann. Allergy 17:380 (May-June) 1959.

A synthetic culture medium for the growth of molds

in the production of mold extracts, which are significantly antigenic and free from nonspecific and irritating substances, is presented. The Center Mold Medium No. 1 is:

Sucrose (chemically pure, ash-free)	40.	Gm.
Ammonium sulfate	2.0	Gm.
Ammonium nitrate	1.3	Gm.
Ammonium citrate	1.0	Gm.
Sodium citrate	2.0	Gm.
Potassium phosphate (monobasic)	0.15	Gm.
Potassium phosphate (dibasic)	0.15	Gm.
Ammonium phosphate (dibasic)	0.2	Gm.
Citric acid	1.0	Gm.
Magnesium sulfate	0.25	Gm.
Calcium carbonate	0.8	Gm.
Trace elements stock solution	1.0	ml.
Distilled water, to make	1000	ml.

The trace elements stock solution contains copper sulfate 0.5%, manganese sulfate 0.5%, potassium dichromate 0.2%, zinc sulfate 0.5%, ferric sulfate 0.1% in distilled water. The methods of preparation of the medium and extraction, concentration, and standardization of the mold extracts are explained. Twenty-one species of molds have been successfully cultured and yielded clinically reliable extracts. A comparative clinical study was done on 100 patients sensitive to molds with extracts of 8 different molds prepared from the medium described, and the Czapek's Medium, another synthetic medium. The patients were skin tested using serial dilutions. All reactions to the extracts prepared by Center Mold Medium No. 1 were immediate and clinically more significant than extracts from Czapek's Medium.

THE PHYSICAL CHEMICAL EVIDENCE FOR ASPIRIN ANHYDRIDE AS A SUPERIOR FORM FOR THE ORAL ADMINISTRATION OF ASPIRIN, Edward G. Garrett.

Physical chemical and kinetic investigations show that aspirin anhydride is a source of aspirin and that it should not produce digestive upset because of salicylic acid production or undissolved aspirin that may adhere to the gastric mucosa and irritate. On oral ingestion, aspirin anhydride should give higher acetylsalicylate blood levels than aspirin alone due to the latter's pH-dependent intestinal absorption. Complete equations are presented for aspirin anhydride's homogeneous and heterogeneous hydrolysis and solubility and the specific equations for physiological conditions have been derived. Procedures have been developed for the assay of aspirin anhydride and all probable derivatives. Thur J. M. PHARM. ASSN., Vol. XLVIII, No. 12, (1959).

DETERMINATION OF WATER IN DRUGS CONTAINING VOLATILE OILS BY MEANS OF THE KARL FISCHER METHOD, M. Sarsunova, L. Cismancova and J. Menkynova (Control Pharm. Lab., Bratislava, Czechoslovakia). *Farmacia, Bratislava*, 1959, 28 (2), 55-57.

The Karl Fischer method is recommended for the determination of water in various drugs of the Czechoslovak Pharmacopoeia containing volatile oils, since it avoids the errors caused in usual procedures by drying the material at 100° to 105°. The water

in the sample is extracted into abs. methanol by stirring for 5 min. in a closed vessel, and is then titrated in the usual way. Thru ANALYTICAL ABSTRACTS, Vol. 6, No. 11, November (1959).

EXPERIMENTS ON THE DETECTION OF PRESERVATIVES USING THE AGAR DIFFUSION TEST, F. Baum and H. Lamm (Inst. Ernährung, Potsdam-Rehbrücke, Berlin, Germany). *Ernährungsforschung*, 1959, 4 (1), 84-89.

The actions of benzoic, dehydroacetic and sorbic acids against the test organisms (*Rhodotorula* spp., *Aspergillus niger*) and baker's yeast were followed by growth on agar plates. The width of the inhibition zones for a given quantity of preservative is specific for each preservative. Thru ANALYTICAL ABSTRACTS, Vol. 6, No. 11, November (1959).

THE REDUCTION OF HYDROLYSIS TIME AND TRYPTOPHAN DESTRUCTION IN THE PREPARATION OF CASEIN HYDROLYSATES, by Vincent S. Venturella, Robert W. Sager and Joseph A. Bianculli (School of Pharmacy, Univ. Pittsburgh, Pittsburgh 13, Pa.), J. Am. Pharm. Assoc., 48, 500

A method was developed for hydrolyzing casein using a procedure which reduces the time requirements of enzymatic protein hydrolysis and minimizes the tryptophan destruction encountered in acid protein hydrolysis. Hydrolysates prepared by this method in twenty-four hours or less are comparable to enzymatic hydrolysates requiring one hundred hours. The method involves the treatment of partial enzymatic hydrolysates with diluted acids.

FURTHER OBSERVATIONS ON LIPID STIMULATION OF BACTERIAL GROWTH. K. Hofmann, W. M. O'Leary, C. W. Yoho, T. Y. Lui (Biochem. Dept., Univ. of Pittsburgh, School of Med., Pittsburgh, Pa.), J. Biol. Chem. 234, 1672-7 (1959).

It was concluded that (a) saturated fatty acid dehydrogenation is not a likely route to cis-vaccenic acid in *Lactobacillus Arabinosus* and *Lactobacillus Casei*, (b) that lactobacillic acid is capable of substituting metabolically for cis-vaccenic acid, (c) that a chain length of 12 carbon atoms is required to endow a fatty acid possessing structure with biotin-sparing activity, and (d) that the short chain fatty acids exert their biotin-sparing effects either by serving as cis-vaccenic acid precursors or by possessing the ability to substitute metabolically for this compound. Thru J. Am. Oil Chem. Soc., 36, 473 (1959).

MICROSCOPIC OBSERVATION OF HUMAN HAIR DYEING PROCESS, S. Preisinger.

In normal conditions of dyeing, the penetrability of dyes depends on the molecule size: the biggest molecule shows the slowest penetrability in the fiber. Simple colourless amines and aminophenols penetrate the hair's keratinic substance and give dark shades with oxygen (oxydative dyes). By nitration of these compounds, coloured products are obtained which are

used as well as for dyeing as for shading hair and are probably fixed by absorption. Microscopic examination of reciprocal interaction between hair and dyes is a source for precious information in the research of new products for shading and modifying natural-coloured and grey-turned hair. Thru PARFUMERIE COSMETIQUE SAVONS, Vol. 2, No. 11, November, 1959, p. 511.

A STUDY OF THE POLYVINYL ALCOHOL-BORATE-IODINE COMPLEX I.-POLYVINYL ALCOHOL-BORIC ACID AS AN INDICATOR FOR IODOMETRIC-IODIMETRIC TITRATIONS, Anthony J. Monte-Bovi and John J. Sciarra.

Solutions of polyvinyl alcohol and boric acid will change color in the presence of iodine. This solution changes from colorless to reddish blue depending upon the concentration of each component. This reaction was investigated in order to determine its application as an indicator for iodometric-iodimetric titrations in place of the usual starch indicator. Several preparations, including iodine, sodium thiosulfate, arsenic trioxide, mercurous chloride, ferric chloride solution, and copper sulfate were assayed by the official method, first by using starch as the indicator, and then through the use of a polyvinyl alcohol-boric acid solution as the indicator. In all cases the polyvinyl alcohol-boric acid indicator gave a satisfactory 4n end point which was comparable to the end point detected by starch. These results indicate that polyvinyl alcohol-boric acid indicator may be used as a replacement for starch in iodometric-iodimetric titrations.

Thru DRUG STANDARDS, Vol. 27, No. 5 (1959). GERMAN PATENT NO. 966,040 (Hans Schwarzkopf), 1957.

Hair Preparation. A great variety of preparations have been recommended to stimulate hair growth. The balanced effect of many factors, such as hormone secretion, vitamin supply, the presence of the heavy trace elements, the equilibrium of the autonomous nervous system and the regional blood circulation and supply seem to be important for a healthy, vigorous hair growth. The last factor—the blood circulation of the scalp—seems to be the key factor in relation to hair growth and the other factors may have only an indirect influence which depends on the blood circulation for their effect on hair growth. Injections with acetylcholine are described in literature as especially effective in the stimulation of blood circulation in the skin. There are, of course, many disadvantages to such a method of injection. These considerations led to a series of experiments which yielded the result that similar good results could be obtained by the local application of preparations which contain compounds from a group of the pantothenic acid tetra alkylammonium bases. The pantothenates of acetyl choline, of carbamineyl choline and acetyl- β -methylcholine are such compounds. Ex.: 12.5% stearic acid; 10% sorbitan monostearate; % polyoxyethylene-sorbitan monostearate; 1.5% acetyl choline pantothenate plus 75% water. ■





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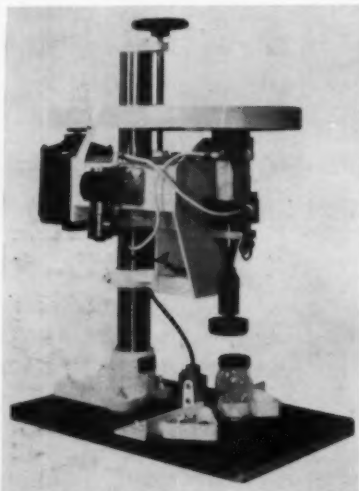
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ly adjustable friction clutch automatically provides uniform cap tightening. It is equipped with a valve which cushions the descent of the capping head preventing damage in event the container is incorrectly positioned. **Scientific Filter Company.**

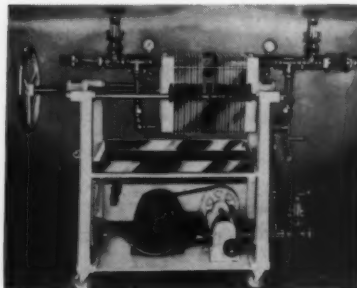
Mechanical locking device for aerosol over-caps

A unique mechanical locking device has been developed which can be incorporated in all types of aerosol overcaps. The device consists of a silver foil label which covers an opening in each cap, where any key may be inserted to unlock and release the cap. After use, the cap may be placed back on the can in a re-locked position to prevent



hazardous or damaging use by children in the home.

The introduction of this tamper-proof aerosol seal makes it possible to better merchandise all aerosol products in department, drug and grocery stores where heretofore they were subject to tampering, especially by children. **Dupli-Color Products Co., Inc.**



Single or double filtration in one operation

A new duplex filter will permit single filtration or double filtration in one operation. It is ideal where some liquids require both primary and polishing filtration and others require a single pass. It is available with either standard or sludge type inlet frames.

One of the most important features is the absence of rubber wash-

ers or gaskets. The filter medium forms a seal around the edges of the filter frames and prevents loss of liquid. It is available in a number of sizes holding from 10 to 100 12" x 12" filtering surfaces. Liquid contact parts can be either stainless steel or nickel plated bronze. **Ertel Engineering Corp.**

Gas sampling valve for uniform volumes

A gas-sampling valve which solves the problem of obtaining reproducible sample volumes for gas chromatography has been announced by Fisher Scientific Company. With this new gas-sampling valve chemists can routinely repro-

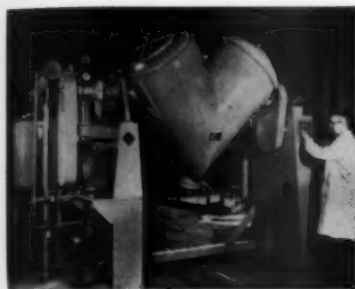


duce gas analyses to within plus or minus 0.2%. Although the valve was developed to increase the accuracy of chromatographic analysis it will be a boon to any gas-analysis system.

A handle controls two mechanically-linked 4-way valves whose Teflon plugs make void-free seals at all 8 ports. Two ports are the inlet and outlet for the sample gas. Two are for the chromatographic carrier gas. The others connect with two loops of stainless steel tubing, in which reproducible volumes of sample are automatically isolated.

The two loops may have the same volume (for a series of measurements of the same component) or different volumes (to measure large and small components with the same accuracy). Fisher Scientific Company.

Packaged solids processor for blending, drying



A new packaged solids processor has been introduced for combining liquid-to-solids blending and vacuum drying. The new combination unit allows precision blending and

drying of a variety of chemical formulations while minimizing materials handling. In operation, dry ingredients are charged and can be tumbled to pre-blend for desired homogeneity under a variety of chosen environments: 1) gentle tumble blending under atmospheric conditions, vacuum, inert or sterilizing gas, or temperatures in the jacket up to 200°F.; 2) intensive blending where pigments, for example, must be intimately dispersed; 3) distribute liquids in solids; 4) disperse liquids under vacuum; 5) blend solids under vacuum; 6) produce tablet granulations; 7) conduct reactions; 8) vacuum dry materials; 9) neutralize static charge in materials by dispersing in the blend a suitable liquid which later can be removed by vacuum drying in the same unit; 10) sterilize materials; 11) coat or lubricate solids, and 12) to dry materials to a fraction of a percent moisture, the unit can be switched over to direct hot gas drying. The Patterson-Kelley Co., Inc.

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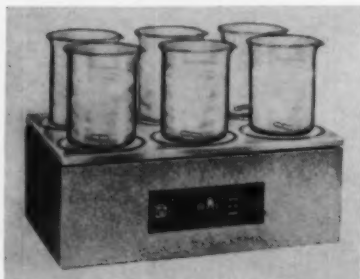


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.. NEWS AND EVENTS ... NEWS AND

Mint Growing Critical in Northwest

Oregon mint growers "face a critical time in their industry," Dan Fry Jr., retiring president, told the Oregon Essential Oil Growers League at the group's 11th annual meeting at Oregon State College. A combination of wilt disease and low prices has put growers in a very "tight squeeze," he pointed out.

Chester Horner, OSC plant pathologist studying ways to control the disease, emphasized the severity of the wilt problem facing the mint growers. More than 6,000 acres of Oregon mint are estimated to be threatened by the wilt and some 4,000 of these acres may have to be replaced soon.

Fumigants seem the best way to control the "exploding" symphyliid problem which is plaguing many mint growers according to H. E. Morrison, OSC entomologist. The insects have resisted efforts to control them with sprays or crop rotation.

Marion Thomas, OSC extension agricultural economist, noted that market prices for mint oil should look up next year and that better prospects for export of mint oil should also help the price situation.

Officers elected at this meeting of the Oregon Essential Oil Growers League were Dale Eisenman, president, Ed Armmon, vice president, James Gossler, treasurer and Iain C. MacSwan, secretary.

DHA Now Available in Industrial Quantities

Dihydroxyacetone (DHA), which could previously be obtained only in research quantities and which sold as high as \$1,000 per pound, is now available in car-

load quantities at about \$8.70 per pound.

A research breakthrough by Wallerstein Company has made possible the use of this fermentation product of glycerine in the manufacture of a wide range of products made by the chemical and drug industries. These include food emulsifiers, plasticizers, fungicides, alkyd-type resins and pharmaceuticals. A new cosmetic product employs DHA as the active ingredient which reacts with the skin to produce a sun tan effect.

Los Angeles S.C.C. Hears Talk on FDA

Gordon R. Wood, chief of the Los Angeles district, Department

of Health, Education and Welfare, Food and Drug Administration spoke to the forty eight members and guests present at the fifteenth regular meeting of the Los Angeles Chapter of the Society of Cosmetic Chemists on January 25, 1960. Mr. Wood outlined the jurisdiction of the FDA as it pertains to the misbranding and adulteration of foods, drugs, devices and cosmetics shipped in interstate commerce.

At this meeting the new officers of the society were introduced. Retiring chairman, Emory Smith, turned the meeting over to these new officers: chairman, Pete Clapp, chairman elect, Oscar Scherr, secretary, Harry Mace, and treasurer, Ben Kapp.



Dr. John M. Longfellow has been installed as 1960 chairman of the New York Chapter of the Society of Cosmetic Chemists. Dr. Longfellow, who is with Colgate-Palmolive has been active as a member and officer in the Chapter since 1955. The 1960 officers are, left to right; Martin Katz, Treasurer, Revlon, Inc.; Agnes Korte, Secretary, Givaudan-Delawanna; John Longfellow; and Saul Bell, Chairman Elect, Chesebrough Ponds'.

Raymond K. Meffen Appointed VP of Hazel Bishop, Inc.

The appointment of Raymond K. Meffen as vice president of Hazel Bishop, Inc. has been announced. He will concern himself with all aspects of the cosmetics company's sales picture. Among executive posts, Mr. Meffen has been general sales manager of Clairol and director of sales for the Lenthieric Division of Olin Mathieson Chemical Corporation. From 1957 to 1959 he was vice president and director of marketing for Hazel Bishop, Inc., to which organization he now returns after 14 months devoted to the development of new products.

N. Y. SCC Dinner Dance Planned for April 22nd

The New York Chapter of the Society of Cosmetic Chemists has planned its annual Ladies' Night dinner and dance for Friday, April 22nd. A full evenings entertainment has been planned at the Diamond Horseshoe, New York City, beginning with cocktails at 6 P.M., dinner (roast beef or lobster) at 7:30 P.M., and dancing and a floor show later in the evening.

Reservations can be made with Mr. Arthur Cohane, Leberco Labs., 123 Hawthorne Street, Roselle Park, New Jersey, CH 5-1933.

Manufacturing Chemists Association Meeting

The 88th annual meeting of the Manufacturing Chemists' Association will be held in White Sulphur Springs, West Virginia at The Greenbrier from June 9th to 11th. Howard S. Bunn, president of Union Carbide Corporation, is program chairman for the meeting. Speakers and program will be announced at a later date.

General Aniline & Film Moves Two Departments

The Dyestuff and Chemical Division, General Aniline & Film Corporation, New York, has moved the traffic and purchasing departments from Linden, New Jersey, to New York City. Mr. John B. Sondey, traffic manager and Mr. S. S. Colman, purchasing agent, and their staffs, will now be located at the Division headquarters 435 Hudson Street, New York 14, New York.



Photo by Ediciones Profesionales, S.A.

In Mexico City last January were (left to right) Sr. Armando Breton, Jr., Gerente, Asociacion Nacional de la Industria de Parfumeria, M. G. deNavarro, editorial director American Perfumer and Adrian Rubio, Beauty Counselors of Cuba, Inc.

New Analytical Section in The Givaudanian

A new section titled "The Analyst's View" was introduced in the February 1960 issue of *The Givaudanian*, house magazine of Givaudan-Delawanna, Inc. Written by Mr. V. D. Johnston, director of the analytical laboratory at Givaudan's Delawanna plant the section will highlight specific aspects of analysis of interest to those active in the chemical analysis of perfumery materials. Mr. Johnston is an active member of both the Scientific and Instrumental Committees of the Essential Oil Association and the American Chemical Society.

Lanolin Plus Debuts "Dew-O-Gen" at New York Press Party

Lanolin Plus has launched their new skin-moisturizing substance, called "Dew-O-Gen" at a press cocktail party held on February 25th at the Plaza Hotel in New York City. Mr. Morton Edell, president of Lanolin Plus, in speaking to the gathering of over 100 members of the beauty and trade press, said "This scientific supplementation of natural sebum, through the use of 'Dew-O-Gen', would start a whole new era in beauty science."

A national advertising campaign will support the introduction of Lanolin Plus Liquid with "Dew-O-Gen", and there will be nationwide retail promotion.

European Tour Scheduled by S.C.C.

The board of directors of the Society of Cosmetic Chemists has voted to sponsor a European tour from August 20-September 9, 1960. The tour will be through France, England, Switzerland, Germany and Spain and will spend three days in Munich attending the first Seminar of the International Federation of Societies of Cosmetic Chemists beginning August 31. Further information may be obtained by writing to Mr. W. H. Higgins, Society of Cosmetic Chemists, 2 East 63rd St., New York 21.

Philadelphia College Course in Parenteral Products

A post graduate course in the preparation of parenteral products will be available at the Philadelphia College of Pharmacy and Science starting July 11th and continuing through July 22nd. The two week course is being offered for the seventh consecutive summer and will consist of lectures, laboratory exercises and field trips. The lectures will cover a variety of topics in the parenteral products field, and in the laboratory a series of small volume parenteral products will be prepared through the cooperative efforts of all who are participating. Opportunity will also be provided for visits to nearby hospitals and manufacturing establishments.

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N.Y. SCC chapter hears two papers on histology

On Wednesday, February 3, 1960, the New York Chapter of the Society of Cosmetic Chemists heard two speakers present current work on histology and "in vitro" maintenance of skin.

Dr. Moire Bradshaw of the Isaac Albert Institute—Jewish Chronic Diseases Hospital, Brooklyn, N. Y. discussed a technique for determining the extent of percutaneous absorption of lipids through normal skin as demonstrated by a fat soluble dye—Sudan Black B. Biopsies of skin to which the dye in lipids was applied were prepared for histologic studies and the presence of the black dye taken as the measure of lipid penetration. Dr. Bradshaw's color slides clearly demonstrated the superiority of cryostatic sections over gelatin sections for this type of work. She further pointed out that animal studies are not applicable to human skin due to the difference in enzymes and skin structure.

The second paper, by Angelica Findley and Ronald Gillette, was

delivered by Miss Findley. She described work on homotransplantation of human and animal tissue at The New York Hospital—Cornell Medical Center. Two salient limiting factors in maintaining organ cultures of skin are: (1) supplying adequate nourishment and (2) preventing de-differentiation to embryonic form. Thin sections of skin were "floated" on a complex nutrient medium for optimum diffusion.

Dedifferentiation was prevented with cortisone acetate in the culture flask. The criterion for successful maintenance of structural integrity is the subsequent successful grafting of the skin to the original donor.

Intact skin has been successfully maintained for as long as four weeks by this method. The organ-culture system may be applicable to a wide range of physiologic problems involving skin, and due to the "flotation" technique, topically applied agents may be studied.

New Aerosol Packaging Line Available

Midwest Consultants, Inc., St. Louis, has added a second complete aerosol packaging line. The new line is a fully automatic straight line 800 series built by The Kartridg Pak Co., Franklin Park, Illinois. Midwest Consultants specialize in filling products which they formulate for private label and name brand marketers.

Fifth Year for Pharmaceutical Research Discussion Group

The Pharmaceutical Research Discussion Group which meets monthly to discuss non-confidential aspects of pharmaceutical product development has begun its fifth year of activity. A round table discussion, which takes place on a topic proposed and discussed by a member of the group or an outside speaker, is part of the regular format of these luncheon meetings. Some of the topics discussed in the past year include: "The Present

Status of Compression Coating", "Relationship between Product Development and Production", "Pharmaceutical Dry Mixing" and "New Developments in Pharmaceutical Aerosols." Members of the group are persons from industry responsible for the research and development of pharmaceutical dosage forms and academic persons representing all of the schools of Pharmacy in the metropolitan New York, New Jersey and Philadelphia area.

Officers of the group for 1960 are: chairman, Harold Sheinaus of Carroll Dunham Smith, secretary-treasurer, Frank Reinish of White, and steering committee, Charles Jarowsky of Pfizer.

Armstrong Glass Plant to Expand

Armstrong Cork Company's glass plant at Millville, New Jersey will be enlarged this spring making the third major expansion at this plant within the past two years.



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New Science Movie Now Available

"Trapping of Free Radicals at Low Temperatures" is a film designed for scientific, technical, industrial and civic groups. The film presents a study of one of the most powerful sources of chemical energy yet discovered—the elusive, highly reactive molecular fragments known as free radicals.

The picture explains the theory of free radicals and describes their behavior. An experiment in trapping and storing free radicals at temperatures within a few degrees of absolute zero by a special process developed at the National Bureau of Standards is shown.

Philadelphia College Observes 139th Anniversary

The Philadelphia College of Pharmacy and Science observed its 139th anniversary on February 18th with a special Student-Faculty Convocation.

President, Dr. Ivor Griffith, conferred honorary degrees of Master of Pharmacy on Gerald S. Pittman of Havertown, Pa., a 1917 graduate of the College and now medical representative for Eli Lilly and Company in the Philadelphia area, and on Orin K. Grettenberger of Lansing, Michigan, director of the Michigan Board of Pharmacy.

Sunbeam Purchases Beauty Supply Equipment Manufacturer

Sunbeam Corporation, manufacturer of portable electrical appliances will acquire John Oster Manufacturing Company, pioneer maker of barber and beauty supply equipment, health appliances and electrical housewares. Sunbeam plans to continue Oster's operations as a wholly-owned subsidiary with no changes in its officers, personnel or facilities. The stock exchange includes all shares of John Oster Manufacturing, companies operating in Mexico and Nassau and related companies. Most of this is held by the Oster family. The total sales volume of these companies for 1959 was an estimated \$35,000,000 with net earnings indicated in excess of \$600,000. The company was founded in 1924 by John Oster, present chairman of the board of directors.

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Pfizer announces 1959 sales, earnings

Chas. Pfizer & Co., Inc. has made public their sales totals for 1959. Their worldwide sales show a 14 percent increase over 1958 and pass the quarter billion dollar mark for the first time. Net earnings were about four percent above 1958 and also hit a new high. 1959 sales were \$253,673,000 and earnings were \$24,863,000.

Expenditures for research during 1959 were increased to \$14.8 million of which \$1.5 million was spent on cancer research. Under a contract with the Cancer Chemotherapy National Service Center of the National Institute of Health, approximately \$1.2 million was reimbursed to Pfizer.

400 members of the research staff of Pfizer have been recently relocated at the new Medical Research Laboratories in Groton, Connecticut. The move consolidates biochemical, chemical and macrobiological facilities in a modern integrated research unit close by Pfizer's Groton manufacturing plant.

New Aerosol Consultant Service in London

Mr. A. Herzka, B.Sc., A.R.I.C., until recently with the Metal Box Company, has established an independent practice as a pressurized packaging (aerosol) consultant, which is carried on under the name of Pressurized Packaging Consultants Ltd., Ashbourne House, Alberon Gardens, London, N.W. 11. Telephone Speedwell 9667/8.

Sales and Earnings Hit New High for Colgate

Consolidated net income of Colgate-Palmolive Company for 1959 was up 20% over 1958 earnings. Edward H. Little, chairman of the board and president, has announced that consolidated sales and earnings of the company for 1959 were the highest in the company's history. Consolidated net income totaled \$25,287,000 and consolidated sales were \$581,982,000, exceeding the 1958 record by \$47,935,000.

Mr. Little credited each of the company's domestic divisions, along with the foreign subsidiaries, for

the increased volume. He also cited the acquisition of the Wildroot Company, Inc. and Sterno Inc. as an important contribution in widening the product base of the Toilet Articles and Associated Products Divisions.

The company's earlier decision to enter the pharmaceutical market has been implemented by the acquisition of Lakeside Laboratories Inc. and S. M. Edison Chemical Company Inc. In addition to the products of these subsidiaries, Colgate will introduce soon in test markets one or two proprietary drugs developed through their own research.

Pharmaceuticals, Inc. to Sponsor "Amateur Hour"

Pharmaceuticals, Inc., through Parkson Advertising Agency, Inc., has bought sponsorship of "Ted Mack & the Original Amateur Hour". This show, which was originated by Major Edward Bowes, will be brought back to television on March 7th, 10:30 to 11 PM, Eastern Standard Time, on ABC-TV.

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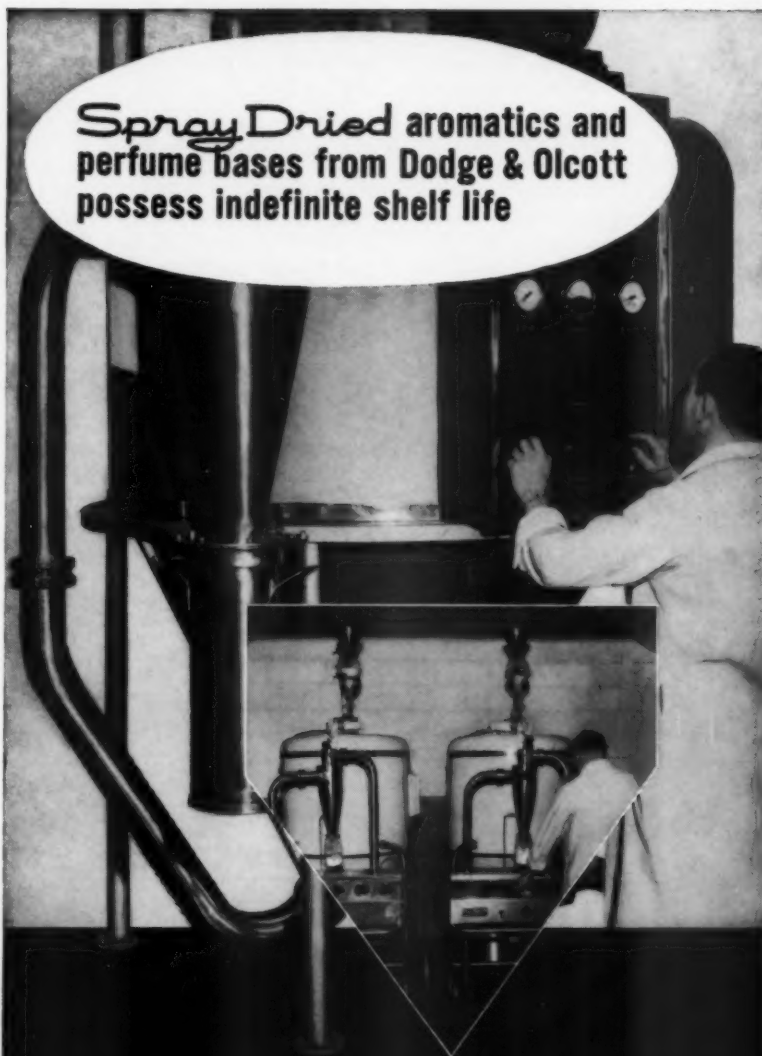
The completed plans for the special Seminar in Aerosol Technology to be conducted by St. John's University College of Pharmacy at the Jamaica campus of the University beginning in March, have been announced by Dr. Andrew J. Bartilucci, Dean of St. John's College of Pharmacy. The program, as arranged by Dr. John J. Sciarra, Associate Professor of Pharmaceutical Chemistry at St. John's and Dr. Francis A. Mina, Technical Director of Lodes Aerosol Consultants, will include a series of six two hour sessions to be held on consecutive Thursday evenings (7 p.m. to 9 p.m.) beginning March 24, 1960 and continuing until May 5, 1960 omitting the session on Holy Thursday, April 14, 1960.

The first lecture on March 24 will cover "Aerosol Principles" and will be given by Dr. Mina; Lloyd T. Flanner, Head of the Aerosol Technical Service Laboratory of the General Chemical Division of the Allied Chemical Corporation will discuss "The Nomenclature and Manufacture of Liquified Gas Propellants" on the same program.

"Liquified Gas Propellants" will be the theme of the second seminar session on March 31. Dr. Samuel Livingood, Assistant Director of the Technical Service Laboratory of Union Carbide Chemical Company will discuss the "Physical and Chemical Properties of the Propellants"; Dr. Fred T. Reed, Aerosol Division Head of Freon Products Laboratory at E. I. du Pont de Nemours and Company, Inc., will talk on the "Toxicity of the Propellants"; Robert C. Webster, Manager of the Customer Service Packaging Laboratory of Air Reduction Company, Inc., will treat "Compressed Gas Propellants"; and David S. Tillotson, Project Leader of Isotron Propellants Technical Service, Pennsalt Chemicals Corporation, will moderate a panel on "Propellants."

The session on April 7 will consider "Aerosol Containers", "Aerosol Valves" and "Aerosol Components." Larry Garton, Supervisor, Non-Food and Analytical Group, Eastern Area Laboratory, America Can Company will speak on the first topic; while Walter Beard,

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Jr., Director of Research for the Risdon Manufacturing Company will speak on "Aerosol Valves". "The Components" will be treated in a panel session moderated by Edmund Budzilek, Chief Chemist of Wheaton Plasti-cote Corporation; and staffed by Joseph Pizzuro, Director of Research for the Precision Valve Corporation and W. W. Wolfe, Technical Service Representative for the Packaging Research Division of Owens-Illinois.

The three part lecture on April 21 will cover "Food Aerosols" by W. Earl Graham, Eastern Manager, Clayton Valve Corporation; "Medicinal and Pharmaceutical Aerosols" by Dr. John J. Sciarra, of St. John's; and "Cosmetic Aerosols" by Victor DiGiacomo, Administrator of the Perfume Laboratories of Givaudan-Delawanna, Inc.

John Hart, Assistant Chief Chemist, Aerosol Plant, Stalford Pressure-Pak, Inc., will speak on "Aerosol Packaging Equipment" at the April 28 session. "Plant Operation" will be covered by Anthony Iannacone, Technical Director of the Fluid Chemical Company, Inc; and a

demonstration of laboratory equipment will be made by Dr. Sciarra and his staff.

The last lecture will cover "New Drug Applications for Aerosols" and will be given by Julius Hauser, Administrative Assistant to the Director of the Bureau of Medicine of the Department of Health Education and Welfare of the U. S. Food and Drug Administration.

The seminar is designed for those in pharmaceutical, cosmetic, food and chemical specialties industry, desirous of gaining more than a fundamental knowledge of aerosols. A certificate of attendance will be awarded to those attending the seminar and a special reception will be held on May 5, the closing evening of the series. The series fee is \$30.00. Advanced registration is required.

Obituary

Luc Descollonges, brother of Jean Descollonges, was killed in a skiing accident on Sunday, February 21st in Chamonix. Mr. Descollonges was manager of the Descollonges Freres plant in Lyon.

Kolmar Laboratories Executives Travelling

John Rix, president of Kolmar International, Inc. recently visited Kolmar de Mexico, S.A., for consultation with Guillermo Trevino, managing director. Ing. Trevino will visit in the States shortly for a refresher course and to contact various firms. Winton Burger, assistant to the managing director, Leslie Hall, of Kolmar of Australia, spent some time in the United States recently taking a refresher course.

Hospital-used Skin Lotion goes on open market

"T.L.C.", a skin lotion manufactured by Polychem Corporation of New Haven, Connecticut, is now being marketed through drug outlets for consumer use. This product was previously sold only to hospitals and was widely used to prevent bed sores. Packaged in a flip-top squeeze dispenser the product is being promoted for body massage to prevent dry skin and soothe minor irritations.

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PERSONALITIES



Louis Brennesholtz

Mr. Louis Brennesholtz, manager of operations for Yardley of London, Inc., was appointed a director of the company. Mr. Brennesholtz fills the vacancy on the board created by the retirement of Edwin B. Millar, former secretary-treasurer. Headquartered at the main Yardley plant in Union City, New Jersey, Mr. Brennesholtz has been manager of operations for the American company for three years.

William V. Reynolds has been appointed director of international operations of the Lehn & Fink Products Corp. In this position Mr. Reynolds will be responsible for the overall direction of the company's branches and sales agencies throughout the free world as well as subsidiary companies with plants in Buenos Aires, Rio de Janeiro, Toronto, Paris, London and Hamburg.



William V. Reynolds

David Garlen has been named director of research for Lanolin Plus. Mr. Garlen, who served as assistant research director for the company since June of 1958, is noted for the creation of "Wash 'n Curl", the first protein-waving shampoo. He has been in the cosmetic industry for the last ten years and was formerly senior chemist in hair products at Revlon Products Corp.



Dr. Harold Schwartz

Dr. Harold Schwartz has been appointed director of toiletries and cosmetics research for the Warner-Lambert Research Institute. Formerly director of Pharmacology for the Revlon Company, Dr. Schwartz succeeds Dr. Donald H. Powers who has retired but will continue as a vice president of the Research Institute in a consulting capacity.

Dr. Schwartz at one time was associate director of the Laboratory of Industrial Hygiene, where he specialized in the biological evaluation and testing of cosmetics products.

At the same time, it was announced that Dr. Martin M. Rieger has been promoted to the new position of director, toiletries and cosmetics laboratories of the Warner-Lambert Research Institute. Prior to joining Warner-Lambert in 1955, Dr. Rieger was research chemist with Lever Brothers.



Mrs. Ida Crawford Stewart

Mrs. Ida Crawford Stewart has been appointed to the position of merchandising coordinator of Coty, Inc. Mrs. Stewart, formerly associated with the Bristol-Myers Co., is corresponding secretary of the Advertising Women of New York.

Thomas Francis Ney has been named assistant sales manager of Lenthic-Kings Men. Mr. Ney was formerly eastern regional manager of the company and prior to that was sales representative for the State of Michigan and special assistant attached to the Chicago office.

Mr. Thomas Miserendino has joined Flam-Haft Laboratories, Inc. as vice president and perfumer in charge of the perfume and flavor departments. He is a member of the American Society of Perfumers and has been associated with the industry since 1939.



Thomas Miserendino

Dr. Charles H. McBurney has been appointed director of research of Rohm & Haas Company, Philadelphia chemical manufacturer. The position was previously held by Dr. Ralph Connor who continues as vice president in charge of research and chairman of the board. Dr. McBurney has served as assistant director of research for the past ten years.



Joseph A. Danilek

Joseph A. Danilek, president of Mary Chess, Inc. and Parfums Schiaparelli, Inc. was recently made a Knight of the Sovereign Military Order of Malta at a solemn investiture ceremony in St. Patrick's Cathedral at which His Eminence, Francis Cardinal Spellman was the Celebrant.

Mr. Danilek was bestowed this honor by a Papal decree from Rome. The Order of Malta is the oldest order of Knighthood in existence, dating back to the days of the Crusades of the eleventh century, and its special purpose is the care of the sick and poor. Mr. Danilek is widely known for his active interest in civic, charity and church affairs.



Miss Mary Vallade

Dr. Joseph P. Simko, Jr. and Mr. Gilbert J. Suarez have been appointed to the Analytical and Instruments Section of Colgate-Palmolive's Corporate Research and Development Department.

Dr. Simko returns to the Colgate staff from Esso Research and Engineering Co. He was a member of Colgate's research staff for several months in 1957.

Mr. Suarez has been serving as a technician on Colgate's research staff since 1955. He recently received his B.S. degree in chemistry from the College of the City of New York.

Jack I. Le Vant has been elected to the newly-created position of executive vice president and general manager of the S. M. Edison Chemical Company of Chicago. Formerly manager of sales, merchandising and advertising, Mr. Le Vant, will now direct the operations of this wholly-owned subsidiary of Colgate-Palmolive Company.

George R. Sivy has been appointed sales promotion manager of Shulton, Inc. He replaces Edmund Jackson who recently was named brand advertising manager. Prior to joining Shulton, Sivy was assistant retail promotion manager at McKesson & Robbins.

Lynn Boland, William J. McCartney and Edward K. Shukur have been appointed product group supervisors, and Mary Louis Casey, Alvin S. Levine and Allan Rosenberg have been appointed assistant product managers, in the further expansion of the product management group at Helene Curtis.

Miss Mary Vallade and Miss Kay Van Norden Netz have been appointed special training representatives for Lenthéric-Kings Men. Miss Vallade has been assigned the western region of the country and Miss Netz the eastern region. The two women are the first of several training representatives who will be appointed to work closely with Lenthéric customers in the area of promotion and fragrance education.

Dr. Robert H. Barth is now manager of research, responsible for the operation of Heyden Newport Chemical Corporation's Garfield, New Jersey laboratories. Dr. Barth has been with Heyden since 1938 and has been research supervisor at the company. He is an acknowledged research authority on the chemistry of pentaerythritols.



Dr. J. Stephan Jellinek

Dr. J. Stephan Jellinek recently joined the perfume staff of Polak's Frutal Works, Inc., Middletown, New York. After completion of his chemical studies at Cornell University and the University of Wisconsin, Dr. Jellinek became associated with the perfume department of P.F.W. in Amersfoort, Holland where he has been active for the past several years.

Wilson F. Fowle, Jr., has been named Wildroot product manager for the Toilet Articles Division of the Colgate-Palmolive Company. Prior to joining Colgate, Fowle served for more than five years with the Benton & Bowles advertising agency, most recently as a senior account executive on package goods products.



Miss Kay Van Norden Netz

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Jack K. Lipson

Jack K. Lipson has been appointed director of advertising of Helene Curtis Industries, Inc. Although Lipson has been advertising manager of the company for some years, this new appointment points up the expansion of the advertising activities of the firm, and the departmentalization of the advertising effort. Aside from the normal advertising functions and departments that will report to him, the newly established package design department will fall under his jurisdiction, as well as all product publicity efforts. Donald H. Hall has been appointed assistant to the

director of advertising. Irving Koppel has been appointed new products manager. He has been associated with Helene Curtis in various merchandising capacities for several years. This appointment is in line with the concentration and specialization of effort by Helene Curtis in areas designed to insure further and future growth of the company.

William P. Claussen has been appointed assistant general sales manager of the New York home office of McKesson & Robbins, Inc. Mr. Claussen, formerly sales manager at the Minneapolis Drug Division, joined McKesson in 1950. He has served as sales assistant, salesman, assistant sales manager and merchandise manager at the company's Minneapolis unit.

Mr. John ter Veer, secretary of Polak's Frutal Works, Inc., was honored recently on the completion of his 40th anniversary with the company. He joined P.F.W. in 1920 in Amersfoort Holland in the accounting department. In 1923 he was transferred to the United States and in 1928 was elected secretary of the company.

Donald H. Francis has been made a vice president of Stepan Chemical Co. Currently general manager of the Maywood Chemical division of Stepan, Mr. Francis will continue to direct this division's operations in Maywood, New Jersey. Mr. Francis joined the Stepan organization seven years ago as chemical engineer and later became director of engineering.

Mark Winter has been named to the sales staff of Reynaud, Ltd. He will work out of the company's headquarters calling on cosmetic and perfume manufacturers in the New York metropolitan area.



Mark Winter



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